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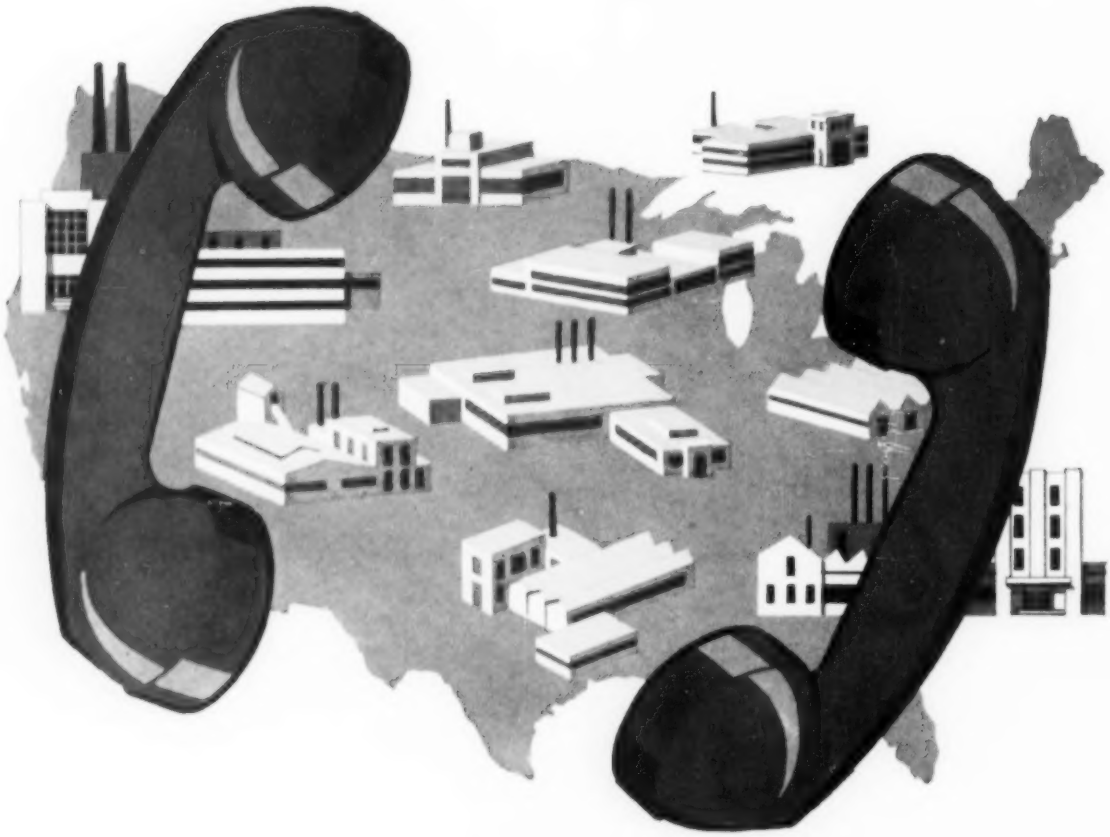

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JOURNAL OF

METALS

VOL. 9, NO. 7

JULY 1957

This issue of JOURNAL OF METALS is in Two Sections:
1—Magazine 2—Transactions Section

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COVER

A seventeenth century charcoal furnace, plus other symbols representing the progress of 300 years, call attention to the second part of "Blast Furnace U.S.A.," which appears on p. 957. Original etching by E. W. Hale; cover by Irene Trivas.

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(Continued on page 1004)

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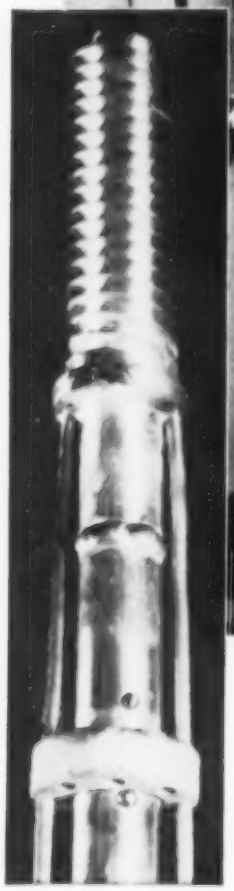

Melvin L. Bleiberg, at Bettis Laboratory since 1952, operates the specially designed cooling system for his unique low temperature in-pile cryostat. Mr. Bleiberg is a member of the Solid State Studies Group, which works on fundamental metallurgical projects at Bettis. He received his master's degree in metallurgy from the University of Pittsburgh under the Westinghouse educational assistance program.

Inset shows sample chamber heat exchanger before being enclosed in an aluminum housing. The coolant gas enters the chamber from the aluminum tubing and circulates through the heat exchanger which is a double-pitch, double threaded screw. The test samples are enclosed inside this heat exchanger.

THE RESEARCH AND DEVELOPMENT OF NUCLEAR FUELS

A Study of Property Changes under Radiation at Low Temperatures

Nuclear fuel materials undergo drastic property changes due to neutron irradiation. Metallurgists at Bettis Atomic Power Division are actively conducting research in this new field of nuclear energy.

One typical experiment is the determination of the property changes of fissionable materials in-pile at low temperature. This unique study is based on previous work done by metallurgists at Bettis Plant* which showed that uranium-base alloys exhibit phase changes due to the influence of neutron bombardment.

In order to study this phenomenon, a special experimental facility had been designed to obtain and sustain low temperature during irradiation of fissionable material. How to remove the heat generated by fissioning of uranium was the major problem in this experiment. M. L. Bleiberg, senior engineer, solved the problem by developing a cryostat in which specimens of fissionable materials could be maintained at temperature of less than −100° C. by means of flowing helium gas cooled by liquid nitrogen.

The first cryostat will be inserted into the reactor at Brookhaven National Laboratory, where it will become a semi-permanent facility to test different samples of nuclear materials. The data obtained from these tests will be used to develop improved fuel alloys for the nuclear reactors now being designed and developed at Bettis Atomic Power Division.

This is only one example of the challenging work conducted here. We welcome inquiries from metallurgists interested in the excellent careers offered by the new and growing nuclear power industry. Please address your résumés to: Mr. M. J. Downey, Department A-172, Bettis Atomic Power Division, Westinghouse Electric Corporation, P.O. Box 1468, Pittsburgh 30, Pennsylvania.

*M. L. Bleiberg, L. J. Jones, B. Lustman, "Phase Changes in Pile-Irradiated Uranium Base Alloys," *Journal of Applied Physics*, Volume 27, p. 1270-83 (1956).

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Next month—Metallurgical Process Development—Weld-Conditioning of Reactive Metals for Nuclear Applications

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SAMPLE IDENT.	C	Mn	Cr	W	Mo	Cu	Fe	Co
12-24-55		2.16	24.36	6.75	3.11			12.90
1- 3-56		2.16	24.35	6.69	3.09			12.88
1-12-56		2.12	24.32	6.73	3.09			12.90
1-19-56		2.14	24.21	6.60	3.07			12.88
3- 9-56		2.14	24.36	6.85	3.12			12.88
3-14-56		2.11	24.40	6.68	3.08			12.92
3-16-56		2.13	24.28	6.57	3.08			12.98
3-20-56		2.12	24.23	6.65	3.09			12.96
4- 9-56		2.10	24.35	6.74	3.08			12.93
4-10-56		2.09	24.37	6.66	3.08			12.95
4-16-56		2.13	24.29	6.65	3.09			12.98
4-18-56		2.13	24.56	6.66	3.10			13.00
5- 2-56		2.11	24.31	6.82	3.08			12.98
8- 6-56		2.13	24.29	6.59	3.08			12.88
8-10-56		2.14	24.34	6.69	3.06			12.91
8-13-56		2.16	24.33	6.59	3.09			12.91
9-28-56		2.18	24.38	6.48	2.94			12.86
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The Fatigue of Metals, *The Institution of Metallurgists,* London S. W. 1, England, 148 pp., approx. $2.45, 1956.—This book features a discussion of fatigue of notches, surface finishes, etc.; structural aspects of aircraft fatigue; corrosion fatigue; and the effect of temperature on fatigue properties. A tabulation of fatigue data on a number of alloys supplements the text. •

Chemistry in the Metallurgy of Iron and Steel, by John Chipman, Phi Lambda Upsilon, Dept. of Chemistry, *Pennsylvania State University,* University Park, Pa., 95 pp., $2.50, 1957.—These are notes of the 31st Annual Priestly Lectures, sponsored by Phi Lambda Upsilon and associated departments of the university. The following topics are covered: oxidation and reduction in liquid steel, Henry's law in metals, metallurgical slags, hydrogen in steelmaking, and dephlogiesticated air in the steel industry. •

The Final Forming and Shaping of Wrought Non-Ferrous Metals, Monograph and Report Series No. 20, by *The Institute of Metals,* 17 Belgrave Square, London S. W. 1, England, 128 pp., approx. $2.50, 1956.—This published record of a symposium held by the Institute of Metals in London, April 1956, consists of eight papers, a general discussion, and indexes by names and subjects. The papers deal with machining properties, deep drawing and spinning of sheet metal, rubber pressing, cold-roll forming of light gage sections, stretch forming, bending and allied operations, and the hot forming of magnesium alloys. The papers are reprinted from the *Journal* of the Institute.

Semiconductor Abstracts—Abstracts of Literature on Semiconducting and Luminescent Materials and Their Applications, Vol. III, edited by E. Paskell, Battelle Memorial Institute, *John Wiley & Sons,* 332 pp., $10.00, 1957.—Sponsored by the Electrochemical Society Inc., this volume (or 1955 issue) contains 1258 abstracts, almost double the quantity of the previous book in the series, and includes abstracts of papers presented at several technical society meetings. In bibliography format and arranged on the basis of materials, the book covers the properties and applications of germanium and silicon and contains sections on carbon, selenium, intermetallics, oxides, arsenates, phosphates, and organics. A final section on theory alone is nearly 50 pp. long. The 1953 and 1954 issues are $5.00 each. •

Deoxidation of Steel by C. H. Herty, Jr., *National Open Hearth Steel Committee of AIME,* 29 W. 39th St., New York 18, N. Y., members $7.00, nonmembers $10.00, various pagings, 1957.—This memorial volume to C. H. Herty, Jr., is a collection of his selected papers which emphasize Dr. Herty's most significant work on the physical chemistry of steelmaking. The papers, generally unavailable today, deal with elimination of metalloids, solubility of iron oxide in iron, deoxidation with silicon and aluminum, and the control of oxide in the open hearth process. In addition, other problems affecting refining reactions and the quality of steel are discussed. The book includes a biographical sketch, a list of Herty's publications and patents, a chapter on the significance of his work, and short biographical sketches of 37 of his co-workers on the Physical Chemistry of Steelmaking Project. •

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Sept. 12—Design Data Evaluation, Economics, Melting and Fabrication
Sept. 13—Corrosion and Fabrication

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For information, contact Dr. H. Margolin, New York University, College of Engineering, New York 53, N. Y. Phone LUdlow 3-6310.

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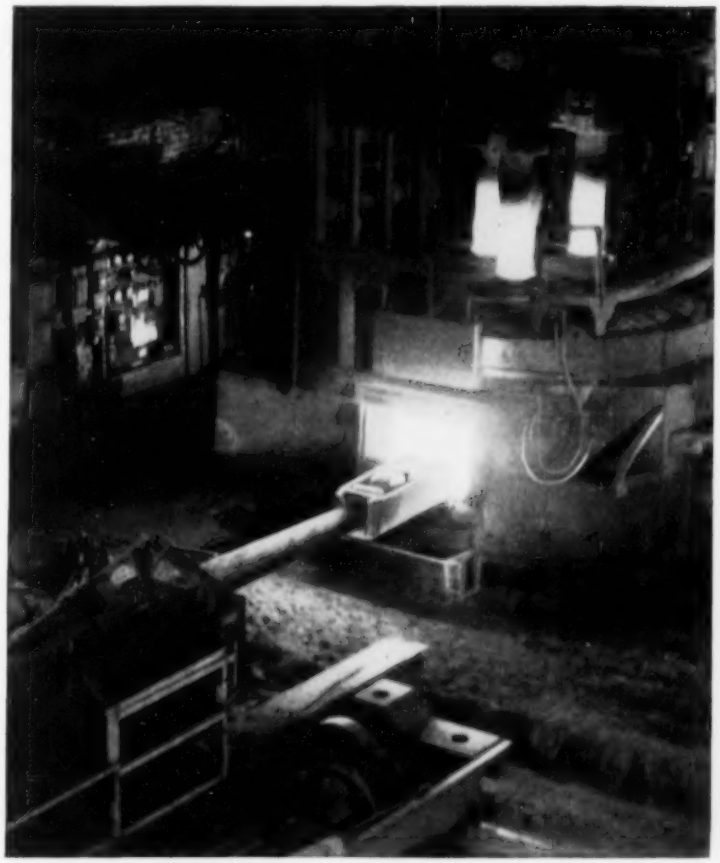

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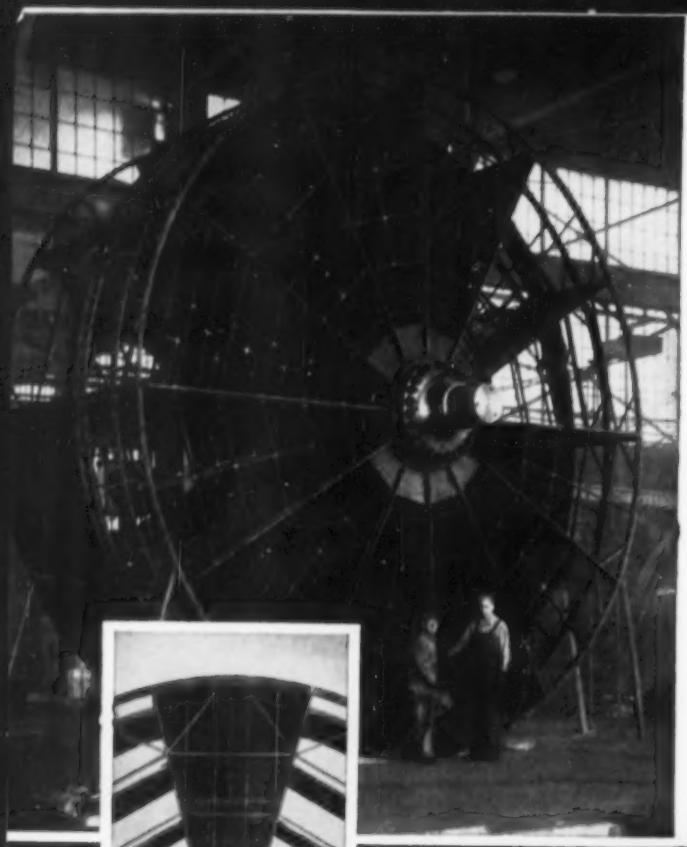

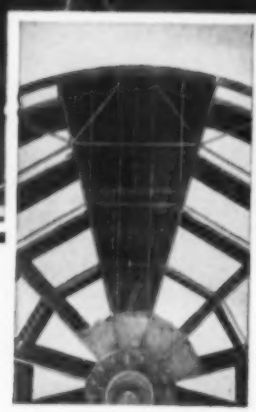

INCREASES CORROSION RESISTANCE. The huge rotor illustrated here is the main structural component of a Ljungstrom Horizontal Air Preheater. Three of these units, designed to serve a 1,900,000 pounds-per-hour-capacity boiler, are among the largest ever manufactured by The Air Preheater Corporation, New York. Each complete preheater weighs 270 tons. The rotor itself—25'2" in diameter and 10'10½" deep—when fully loaded with the heating element, weighs about 390,000 lbs.

Because sulfur in the gases combines with moisture—sulfuric acid is formed when the temperature drops below the dew point at the "cold" end of the rotor—corrosive action can be highly severe in this area. Corrosion means maintenance.

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As a consequence, USS COR-TEN Steel is specified for the cold end heating elements and containing baskets on all conventional boiler applications. Where unusually severe corrosion is anticipated, USS COR-TEN Steel is also specified in all or part of the rotor including diaphragm plates, bar stock, rim angle and filler plates.

INCREASES DURABILITY, REDUCES COST. Tote boxes have to take quite a beating. Used for handling, storing and shipping automotive and other parts, they must be able to withstand plenty of rough treatment.

That's why the "Hamlintainer" shown here—a *collapsible* tote box that sets up and folds flat in less than 20 seconds—is now built entirely of USS COR-TEN Steel. COR-TEN Steel's greater strength, 50% higher than carbon steel, makes it possible to build the "Hamlintainer" up to 100 lbs. lighter than carbon steel units, yet so strong and rigid that it will withstand long and rugged service and is not susceptible to bending and distortion. The fact that the COR-TEN Steel ends and sides have the stamina needed to permanently maintain their shape is of utmost importance. It means that throughout its long life the box will always be easy to set up, fold and stack flat.

As compared to the metal construction used in an earlier design, USS COR-TEN Steel makes the "Hamlintainer" not only stronger, more rigid and more durable but also less costly to produce, according to the manufacturer, Hamlin Metal Products Co., Akron, Ohio.

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High-strength USS COR-TEN Steel pays off in equipment like this

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USS COR-TEN Steel is distinguished by its superior resistance to atmospheric corrosion—4 to 6 times that of carbon steel, 2 to 3 times that of copper steel.

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Thus, when used to directly replace carbon steel, USS COR-TEN Steel will materially increase the strength and durability of vital parts *without* increasing their weight. Or it can be used in thinner sections (1) to reduce weight without sacrificing strength or (2) to increase the capacity of equipment without increasing gross weight or the power required to move it.

You will find our 174-page "Design Manual for High Strength Steels" extremely useful in applying USS COR-TEN or our other High Strength Steels, USS MAN-TEN and USS TRI-TEN "E" to your product. For free copy, simply write on your company letterhead to United States Steel, Room 2801, 525 William Penn Place, Pittsburgh 30, Pa.

For more information, Circle No. 383

INCREASES STRENGTH, SAVES WEIGHT. Shown here hauling a 117-ton transformer, this 150-ton-capacity trailer—built by Columbia Trailer Company, Vancouver, B. C., for the Arrow Transfer Company of that city—is the largest trailer ever built in Canada.

This 80-ft.-long trailer is constructed almost entirely of USS COR-TEN Steel. It is about 25% lighter than if it had been built of structural carbon steel. Specifically designed for handling transformers of giant size, it has a depressed center deck which makes loading and unloading easier and keeps center of gravity low to prevent danger of upset.

Says the manufacturer: "We have found that when a trailer is made from structural carbon steel there is a greater possibility that it can be permanently damaged from overloading than in the case of a similar unit made from high strength steels. That's why, in designing trailers of this type, we always use USS COR-TEN Steel. This construction gives us the high strength needed, plus excellent corrosion resistance and freedom from excess weight—all very important in equipment like this."

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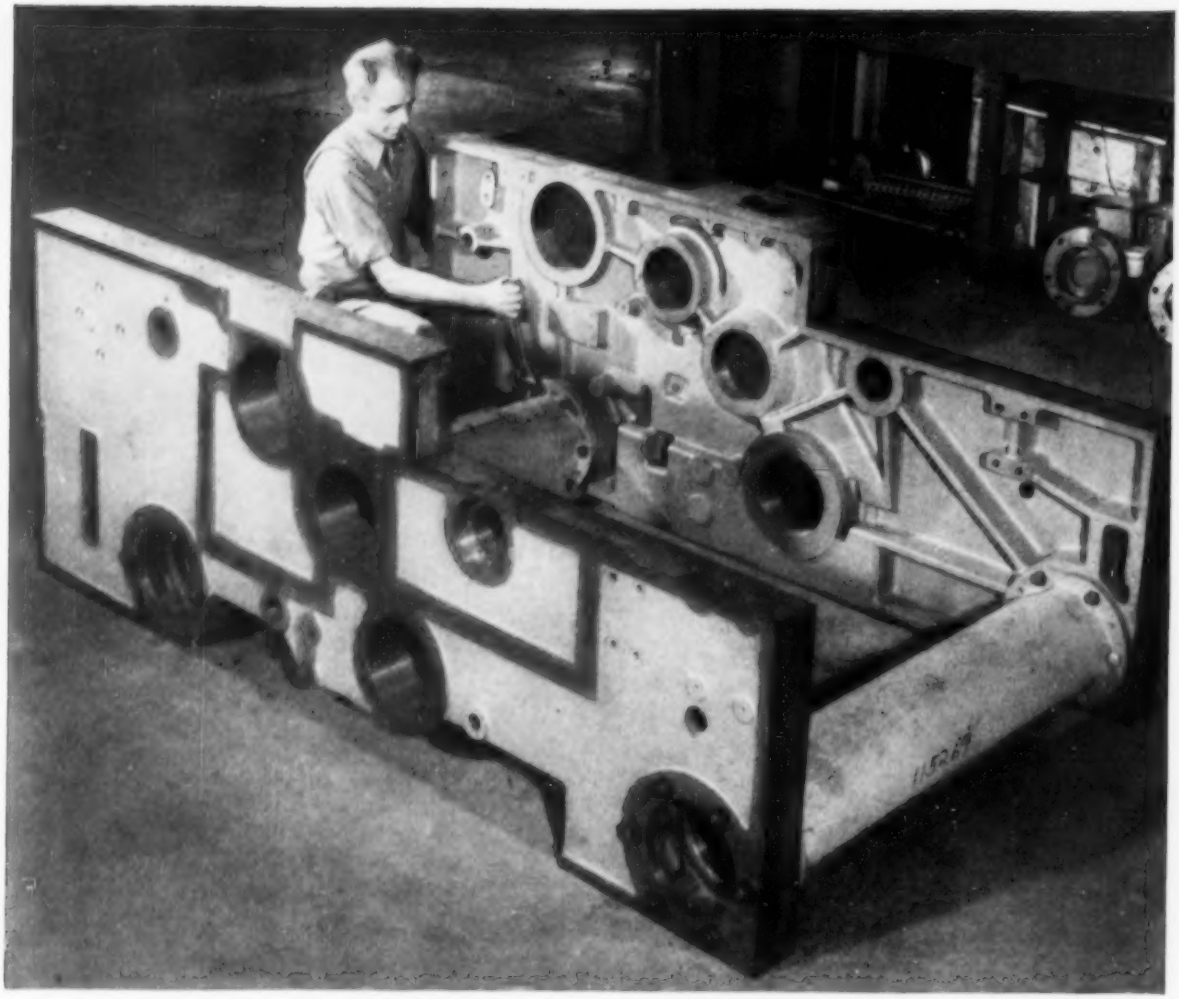
USS HIGH STRENGTH STEELS

USS MAN-TEN · USS COR-TEN · USS TRI-TEN

UNITED STATES STEEL



1300 pound cast frame for large high speed paper-making machine. Nickel added to cast iron eliminated deformations caused by internal stresses. Frame cast by L. Brayton Foundry Co., for St. Regis Paper Company, East Providence, R.I.

Deformation problem in heavy cast frames straightened out with 1.00% nickel cast iron

St. Regis Paper Company had a problem machining large cast frames for high speed multiwall paper-bag-making machines.

When frames were cast, differences in cooling rates between thick and thin sections caused wide variations in the "as cast" microstructure. And trouble began.

The base iron, adjusted to give dense, wear-resisting structures in heavily bossed areas, was chilling in ½" web sections. Result: internal stress, deformation during machining and serious misalignment in finished frames.

Heat treating? Not quite the answer. It would boost the cost of the casting 25%. There was another way. An easier way. A sure way.

1.00% nickel addition solves deformation problem.

St. Regis engineers working with the foundry decided on a 1.00% nickel addition to the base iron. It solved the problem. The nickel brought about uniformity of microstructure. It reduced chill and eliminated the stresses resulting from it.

This increased stability paid off in another way: it saved valuable production time. With nickel cast iron, machining operations keep right on going — without interruptions for stress relief or danger of hard edges. The nickel also makes the frame easier to machine to the tolerances required.

Do you have a metal problem? Let's talk it over. In the meantime, a booklet, "Nickel Alloyed Cast Irons" may give you a clue to the solution. Just write.

INCO THE INTERNATIONAL NICKEL COMPANY, INC. 67 Wall Street New York 5, N.Y.

Steel in Sweden–the Stora Story

New, and possibly revolutionary, developments in steelmaking are taking place at the Stora Kopparbergs Bergslags A/B, Domnarvet, Sweden, and they are taking place fast. The Wiberg-Soderfors process for producing sponge iron in electric furnaces received much attention earlier this year (JOURNAL OF METALS, February 1957). A process for producing open-hearth quality steel from high-phosphorus hot metal in a rotating oxygen converter—called the Kal-Do process—is reported on p. 972 of this issue. The latest development called the Stora Powder Steel Process, produces rolled steel directly from pig iron. Desiliconized pig iron is granulated and mixed with about 15 pct high-purity iron ore concentrates, packed in a box and decarburized at 1100°C. The box of granules is then run on a standard rolling mill between 1100° and 1200°C, producing a clean, rolled steel. A rotary Kal-Do furnace is used for the preliminary desiliconizing operation.

National Research Expanding in Many Directions

National Research Corp., prime developer of vacuum metallurgy in this country, is turning its research and development activities into many new fields. Among these are continuous metallizing of plastic sheeting, textiles, and paper, which might become competitive with foil; continuous coatings of steel at high speeds, providing corrosion resistant coatings by vacuum deposit techniques; and a rather deep plunge into the field of zirconium production through the Columbia National Corp., a subsidiary jointly owned with Columbia Southern Chemical Corp.

Developments in Vacuum Technology

Among the many new developments in vacuum technology are a new high vacuum furnace, designed for continuous operation at 2200°C at a vacuum of 0.01 to 0.05μ, built by R. D. Brew & Co. of Concord, N. H. Also, Riehle Testing Machines has developed a device for making autographic optical creep measurements on reactive metals in vacuum furnace, to be on the market soon.

Titanium: Ahead of Schedule

Announcements by the major titanium producers of price cuts amounting to $0.50 per lb on sponge and $0.50 to $1.90 per lb on mill products proved recent estimates of the price trend of this important metal too conservative. Estimates at last year's Titanium Symposium showed that by mid-1957, sponge should be just under $3.00 (current price, $2.25); billet, around $7.50 (now $6.90); and commercially pure sheet, $13.50 (now $13.10). Long range predictions are for $1.28 per lb for sponge and a $2.00 to $3.00 range on mill products, indicating a serious bid by titanium for many nonmilitary applications.

New Desulfurization Process

Battelle Memorial Institute reports a new process for the continuous desulfurization of iron. Molten iron and caustic soda are fed continuously into an apparatus where the materials are intermixed, and desulfurized metal and caustic slag flow continuously from the apparatus into ladles. It is also reported that an oxygen lance inserted into the chamber will remove substantial amounts of the silicon present at the same time sulfur is being eliminated.

EDITORIAL

The Engineering Graduate and Military Service

Summarized from an article by W. T. Cavanaugh, Executive Secretary of the Engineering Manpower Commission of Engineers Joint Council.

THE primary objective of military manpower procurement programs is, obviously enough, to obtain the manpower necessary to meet the requirements of military manning tables.

What is the military obligation? Simply, that every man between age 18½ and 26 who enters the armed forces after Aug. 9, 1955, has a total military service obligation of either six or eight years, depending on the way he decides to discharge it.

Deferments for Graduate Work

Qualified graduate students are permitted, under Selective Service regulations, two years deferment for the masters degree, and four years for the Ph.D. Many graduate students, and undergraduate students for that matter, are currently not called, simply because they are not yet needed by the local board. This kind of delay for academic work may, in the future, become precarious, for reasons that will emerge later.

It is possible to enlist in any of the services for a minimum of three years. From the point of view of the engineering graduate, this is probably the poorest choice, as there are practically no engineering jobs for enlisted men in the armed services. The vast amount of engineering work required within the armed forces is done by civilian engineering employes.

Graduates can take an engineering position in industry, government or education, and perhaps qualify for occupational deferment, which may continue indefinitely under the proper conditions, or terminate, perhaps with qualification for entry into the six months Critical Skills Program.

Occupational Deferment Program

To qualify for occupational deferment, a person has to have, among other things, a critical skill (all engineering skills are critical) and be employed in a critical activity. A list of the critical occupations and activities is available, with other detailed information on this subject, from local boards of the Selective Service System.

Occupational deferments are normally given for a specific period, and are, theoretically, renewable in indefinite sequence. However, local boards want, at some time, to terminate these deferments to ensure that a person is not deferred to a point where his induction, though legally possible until age 35, is impractical for reasons of age, dependency status, hardship, etc. The six months Critical Skills Program was put into the Reserve Forces Act of 1955 and implemented by executive orders in early 1956 to provide a practical and workable solution of the previously unyielding problem of the need for selectivity vs the theoretical need for equity.

The Six-Month Critical Skills Program

The basic requirements for the Critical Skills Program are a 1A classification, and a critical occupation in a critical activity. Unlike occupational deferments (in which the employer is the requesting agent), the registrant himself applies for the Critical Skills Program by a letter to his local board. *This may be done even after notice of induction is received.* Such application automatically suspends all other procedure pending its processing. Upon acceptance, the registrant must, within 30 days, enlist in a Reserve Program. He will be called for six months of active duty for training, after which he is transferred to the Ready Reserve.

Regulations provide for the continuous, automatic screening of the Ready Reserve, so that persons with critical skills, who have these statutory obligations to serve in the Ready Reserve, are actually retained only if requirements exist for their skills, or if they had an overriding military specialty. Persons with critical civilian skills who are in excess of requirements are to be transferred to the Standby Reserve. The exception would be an engineer who is also, say a jet pilot, or possesses some other critical military specialty. It is anticipated that persons with critical skills (whether Critical Skills Program people or two-year service people) will spend relatively brief periods in the Ready Reserve.

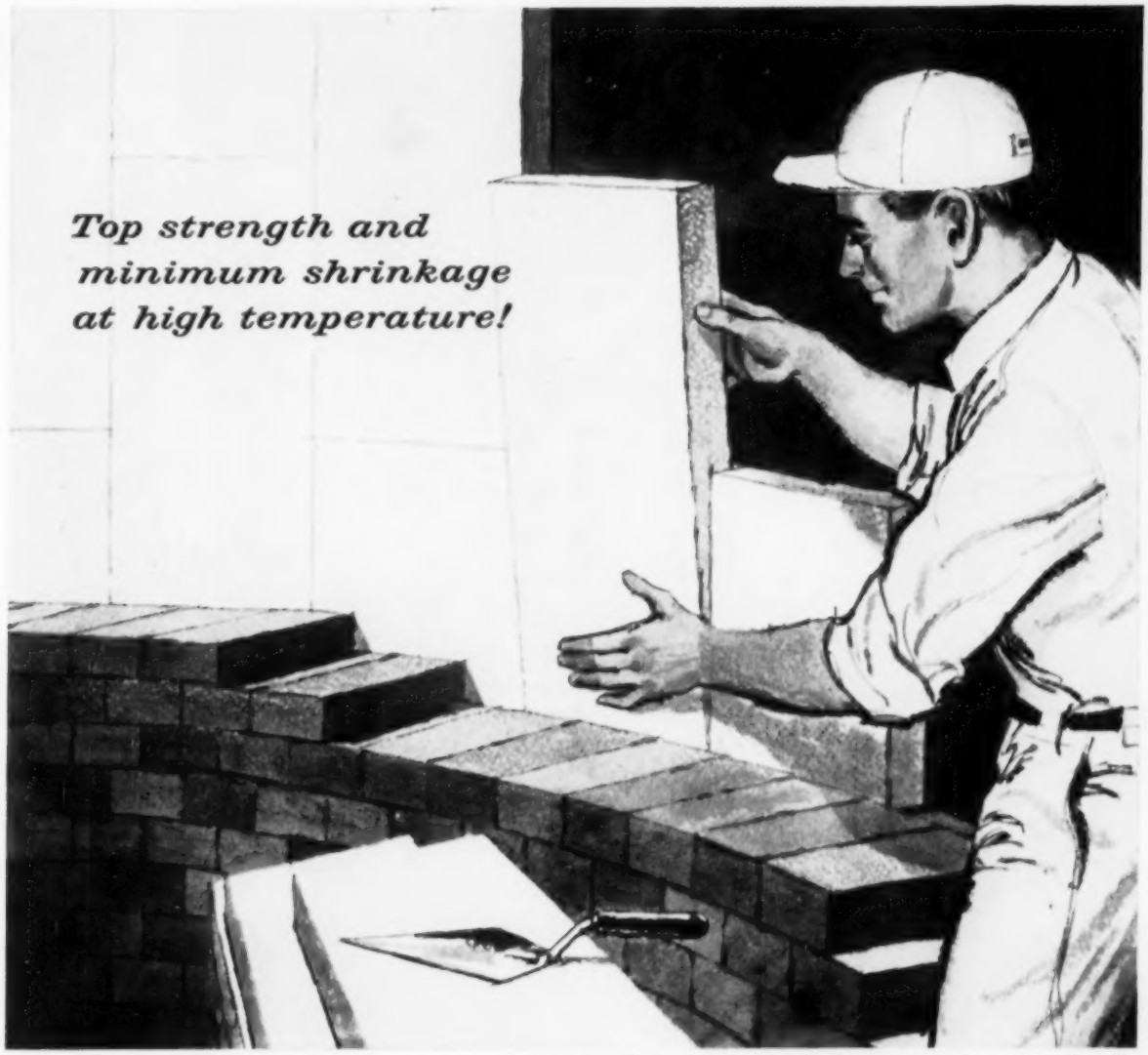
The General Six-Month Program

The purpose of the six-month Reserve Training Program for men under 18½ was to *beef up* the Ready Reserve with younger men on a voluntary basis. After a year of trial, the Program was not meeting its goals, and as a result, the general Six-Month Program was opened to men between 18½ and 26.

There are two important differences between the general Six-Month Program and the Critical Skills Program. First, the general Six-Month Program is a quota program. That is, it will be opened and closed as men are needed. As more men enter the Six-Month Program, the number of men available for military service should be reduced, thereby reducing the chances of obtaining Selective Service deferment for graduate work, etc., because men are not needed. But the Critical Skills Program is a continuing one. Application for the Critical Skills Program can be made even after receipt of notice of induction; this is not so in the general Program. The screening process applies to all personnel, regardless of method of entry into the Reserve Forces.

As is generally known from much public discussion earlier this year, the Army National Guard now has an enlistment program very similar to the U. S. Army Reserve. The Army National Guard is considered part of the Ready Reserve; it is not clear that the automatic screening process will apply to the Guard. Even if the various state governors are sympathetic to it, it would depend on individual request and present a very difficult, decentralized administrative problem.

Questions recent graduates may have regarding the draft situation should be addressed to Mr. W. T. Cavanaugh, Secretary, Engineering Manpower Commission, 29 W. 39th St., New York 18, N. Y.



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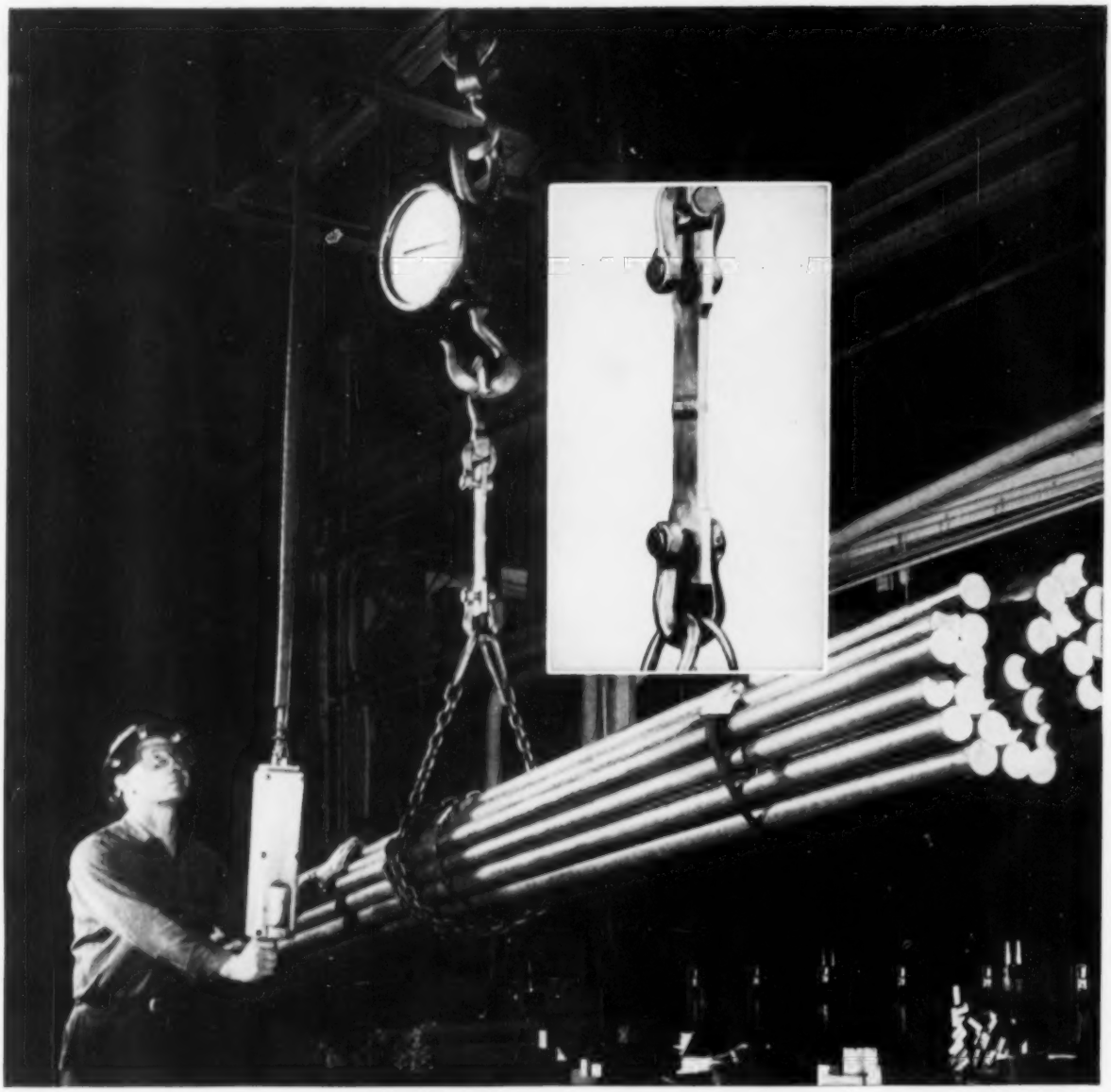

Weld Strength! Magnesium has 95% weld efficiency

In the picture above, a small arc-welded magnesium bar is supporting a load of four tons. This is one example of what we mean by "strong" when we say magnesium is light but strong. Its weld efficiency, the relationship between the strength of the parent metal and a welded joint, is very high. AZ31B magnesium alloy plate, tensile strength 35,000 psi, has a welded joint tensile strength of 33,000 psi, or a weld efficiency of 95%!

Magnesium can be arc welded, gas welded, or welded by electric resistance (spot, seam or flash). Magnesium plate ½" thick can be joined by arc welding in one pass. It can also be readily joined by most any other method: riveting, bolting, screwing, adhesive bonding and self-fastening devices.

These facts spotlight just one of the many reasons magnesium does a better job in many fabricated metal products. For more information, contact your nearest Dow sales office, or write to us. THE DOW CHEMICAL COMPANY, Magnesium Department, Midland, Michigan, Dept. MA1403P.

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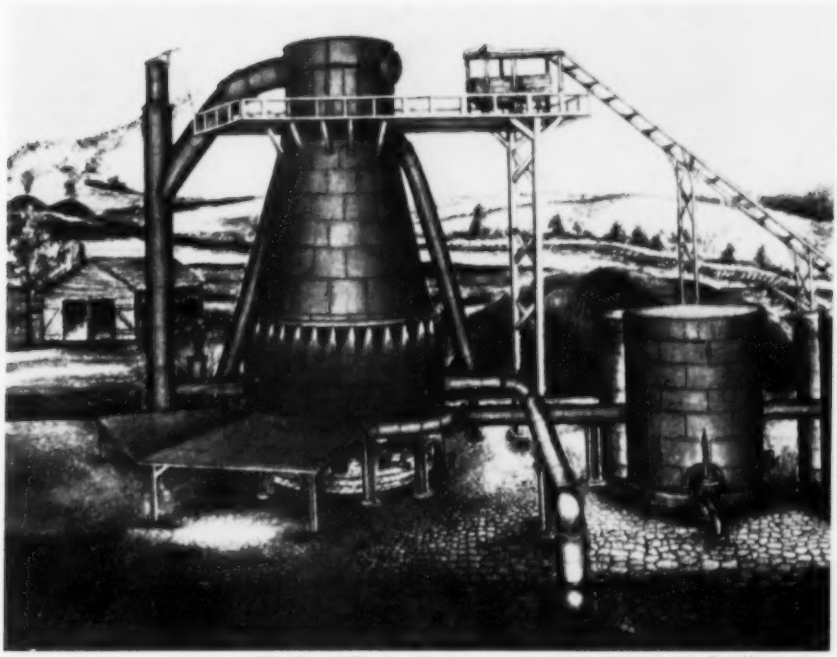


The David Thomas Co. furnace located at Catasauqua, Pa. Illustrations by E. W. Hale.

BLAST FURNACE U.S.A.

II. The Age of Mineral Coal

1750 to 1850: The scene shifts westward across the Alleghenys to the young town of Pittsburgh; charcoal gives way to mineral coal as furnaces grow larger and the blast is heated; above all, Pennsylvania iron is adding strength to the new Nation and building an empire in the West.

by M. O. Holowaty and C. M. Squarcy

TWO names are inseparably associated with the Pittsburgh iron industry—those of George Anschutz and John Hayden.

The iron ore deposits of southeastern Pennsylvania were found and developed early in the 18th century, and several forges and furnaces were built and operated. It was only after the revolution that the iron men crossed the Susquehanna River, built furnaces in the neighborhood of ore deposits of Franklin County, then along the Juniata River and its valley. Juniata Iron found a ready market in the Pittsburgh area. However, difficulty in transporting it across the Allegheny Mountains induced iron masters to seek ore and plant locations in the Pittsburgh district. One of the first to reach this area was George Anschutz, who migrated to America in 1789 at the age of 36, reaching Pittsburgh in the fall of 1790. Anschutz immediately set out to organize an iron company in Pittsburgh, and was able to interest some residents in his project. After being informed of an iron ore deposit located only three miles out of town (on the site of the present 26th St. and the Allegheny River) he decided to build there his blast furnace and foundry plant. Total cost of the installation was $300.

When the furnace was put in operation, Anschutz, now known as Red George, found that the ore deposit was only an insignificant one and would last no more than a few weeks. A new deposit was found on Roaring Run, in the southeastern corner of Armstrong County. Anschutz transported some of this ore to the mouth of Two Mile Run, where it was loaded on horse wagons and transported to the furnace. In this way, he operated off and on for several months, until the furnace was blown out in 1794.

After working for a year as plant manager for John Probst at Langhorntown, he built the Hunting-

M. O. HOLOWATY and C. M. SQUARCY are Chief of Raw Materials Research and Manager of Iron Production, respectively, at the Indiana Harbor Works of Inland Steel Co., at East Chicago, Ind.

ton blast furnace in the county of the same name for Mr. M. Massey and Judge John Gloninger. This operated successfully, and he finally acquired one quarter interest in the plant. Anschutz retired in 1833, and died four years later in his beloved Pittsburgh.

While it was Anschutz who built the first blast furnace in Pittsburgh, the credit for being first to reduce iron ore goes to the Englishman John Hayden of Fayette County. In the summer of 1790, while searching for limestone, he found a deposit that closely resembled the bluish limestone shale of New Jersey, in the vicinity of the present city of Uniontown.

To convince himself of the find, he built a small kiln. After burning the stone for some time, he noticed that the particles fused together and had metallic luster. An examination convinced him that he had found iron ore and not limestone.

Hayden decided then to produce a sufficient amount of iron for a casting, by reducing the ore on a blacksmith forge. A blacksmith in Connelsville took interest in Hayden's problem and ran the experiment which produced several pounds of good grade iron. Hayden now knew that he had struck an iron ore deposit of major significance and decided to exploit it. Carrying his piece of iron, Hayden set out for Philadelphia with the intention of persuading his uncle, John Nicholson, the comptroller of the Commonwealth of Pennsylvania, to invest money in the venture.

Nicholson did not fail his nephew, and in 1791 Hayden was able to lay foundations for his furnace at Fairfield, about seven miles south of Uniontown. Construction was completed and the furnace blown-in in the summer of 1792. Hayden operated the furnace profitably for several years, and then sold it to Oliphant Brothers.

Although Hayden had a good start over his competitors, obtaining the necessary funds delayed him considerably, and before he was ready to produce, two other furnaces were already in operation. The first furnace west of the mountains was built by William Turnbill and Peter Marmie of Philadelphia, on the south side of the Jacobs Creek. The furnace was blown-in Nov. 1, 1790, and soon established an excellent reputation as part of the Alliance Iron Works.

The second furnace of Western Pennsylvania was built a few months later at Dunbar, by the famous Colonel Isaac Meason. Both furnaces were frequently called upon to work for the Government. Major Isaac Craig, deputy commander of Fort Pitt, ordered from Alliance Iron Works two tons of 6 lb solid shot, one ton of 3 lb shot and one ton grape. These were sent to Fort Washington (Cincinnati, Ohio) and used by General Wayne in the Battle of Fallen Timbers against united Indian tribes on the Maumee River. This encounter can be regarded as the first victory for Western Pennsylvania iron.

About the end of the 18th century, the future of the charcoal blast furnaces appeared bleak, in spite of the thriving market, because of the shortage of fuel. Fuel was available, but it had to be transported over increasing distances, which eventually brought the price of iron to the point where it was cheaper to buy iron imported from Europe than to use the domestic product.

Mineral coal furnaces—Early failures

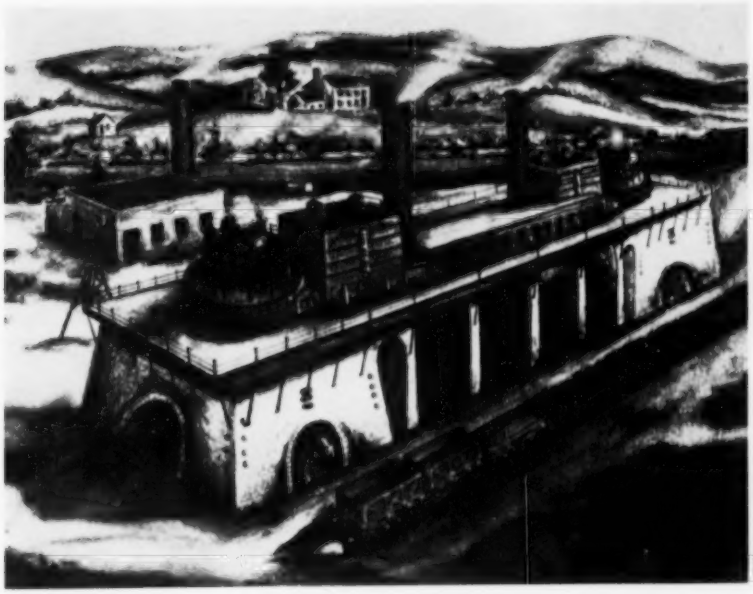
The first step to remedy the fuel situation was to reduce the consumption of charcoal by mixing it with mineral fuels which were readily available. It is almost impossible to determine when the first attempt in this direction was made, but it appears that the old Sterling charcoal furnace was among the first. The furnace was a small one, 5 ft diam at the bosh and 25 ft high. It was built, owned, and operated by the family of Townsends who were related to the Leonards of Saugus fame. The original furnace was located two miles north of the Sterling Iron Works in Warwick Township, Orange County, N. Y. It was apparently around 1770 or 1780 that small amounts of coal were added to the furnace burden. After many experiments, it was decided that coal from Schuylkill County, Pa., was well suited for the one tuyere, cold blast furnace if it did not exceed 1 bushel per 14 bushels of charcoal (approximately 10 pct by weight). This amount, however, was not enough to save the furnace, which was blown out around 1800. Experiences at Sterling profited other ironmakers in similar situations. In most cases, when coal was mined in the vicinity of the furnace (West Virginia, eastern and western Pennsylvania, southern Ohio, etc.) small amounts of it could be added without difficulties, while larger amounts tended to reduce the temperature of the metal.

One of the most interesting operations, from a historic point of view, is that of the Mill Creek Raw Coal Furnace of Master David Grier. His furnace which measured 7 ft across the bosh and 27 ft in height, and stood on Mill Creek, three miles north of Youngstown, Ohio. It was originally built in 1825 as a charcoal furnace. A few years later, difficulties of charcoal transportation and shortage of manpower in the charmaking department induced Master Grier to burden the furnace with bituminous coal mined in the vicinity. After a short experimentation, Grier developed his own furnace practice, according to which the charcoal burden was gradually replaced with bituminous coal until iron in the hearth solidified. The furnace was then slowly blown out and the furnace contents removed. The solid metal was sold as pig, while the sponge iron from the furnace shaft was sold to local forges at a premium price.

After a few years, about 1833, even this practice proved unsatisfactory, since Grier's customers found little attraction in his slogan of *Coal Flame Superior Iron*. The furnace continued to attract sightseers because of an "enormous yellow flame burning at its mouth and visible at night for miles." The Mill Creek furnace was rebuilt to a 9-ft-diam, 30-ft-high unit equipped with hot blast apparatus in 1839, and kept producing small amounts of iron until 1855, when it was finally abandoned.

Starting in 1825, several midwestern furnaces tried peat in the blast furnace. The last and most widely publicized attempt was made by the Lake Superior Iron Company on its 9x45-ft peat furnace at Ishpeming, Mich., in 1873. This method proved uneconomical, although the furnace was known to be still in operation in 1874.

The most unorthodox approach was taken by William S. Scollard, manager of the Springfield hot blast charcoal furnace located at Springfield, Mercer County, Pa. The furnace, which measured 9 ft across the bosh and 35 ft high, was built in 1837. The furnace was burdened on several occasions with dried wood (specie unknown) to relieve the local shortage of charcoal. The exact data on this furnace



An early American coke furnace, similar to many which appeared in Pennsylvania between 1830 and 1850.

are not available, but apparently it continued to operate on the charcoal-wood burden for several years. The serious shortage of charcoal, and later on also that of high grade iron ore, brought about abandonment about 1850.

The coming of anthracite

The use of anthracite in the blast furnace was already established by the end of the 18th century. It was limited, however, to a small number of furnaces located in the anthracite region of Pennsylvania, and to rather insignificant amounts of the material, which was used together with charcoal. The proportion between charcoal and anthracite was usually maintained between 10:1 and 4:1: Amounts larger than 25 pct considerably impaired the performance of the blast furnace and caused the iron to run cold.

Numerous instances of the partial anthracite burden can be found in the literature. Among the oldest reference to *cheaters*, so called for substituting anthracite for a portion of charcoal, is a mention of the Green Mountain Iron Co.'s blast furnace manager Royal Blake, who ". . . every 14 or 16 bushels of charcoal would add a bushel or two of anthracite." Blake's furnace, located in Brandon Village, Vt., was relatively small, its dimensions being 7½ ft across the bosh and 21½ ft high. It was rebuilt around 1854 and converted to a hot blast charcoal and anthracite furnace. The conversion, however, was not successful and the furnace was finally abandoned in 1855.

One of the best known charcoal-anthracite furnaces was the Franklin charcoal furnace of the New Jersey Franklinite Co., built in 1770. It had a 6½-ft bosh and measured 20 ft in height. Small amounts of anthracite were being added in 1805. The amounts were gradually increased until they reached approximately one third of the total fuel burden in 1847 after the furnace was converted to hot blast. The Franklin furnace was used in 1854 for an experiment which was unique in the history of the American blast furnace. The metal burden of the furnace was changed to high zinc iron ore in an attempt to produce both metals from local deposits simultaneously. The experiment failed and the stack had to be rebuilt completely.

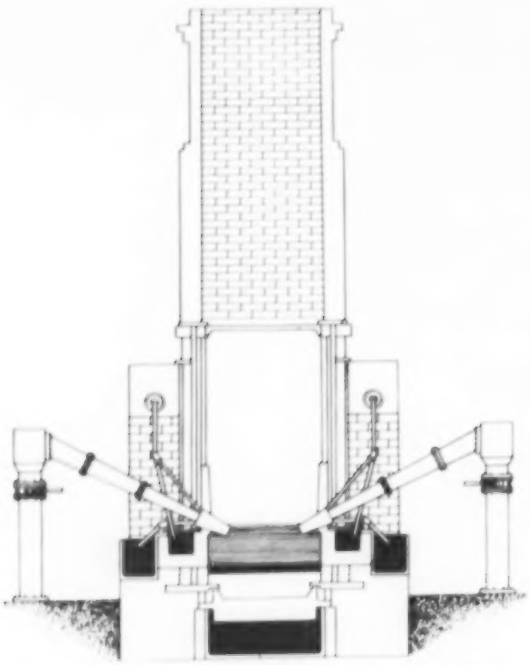
Another attempt to use anthracite was made at the Pioneer furnace owned and managed by J. W. Atkins and his brother, Samuel. The furnace which was built in 1837, was in constant trouble and a notoriously poor producer. Hanging and slipping of this furnace became a byword of the Pennsylvania ironmakers. Fortunately enough, the situation was of short duration. Beginning in 1840, the performance of this first American furnace operated along the rules established by Thomas, began to improve considerably, until by 1850, it was regarded as one of the most efficient furnaces in the country.

The increasing shortage of charcoal induced the Pennsylvania legislature to pass in 1836 an act "for the encouragement to manufacture iron by mineral fuel." The act gave the governor authority to charter companies with ample powers in regard to the stock and quantity of land which might be required. It was probably this act which stimulated the widespread investigations of the applicability of anthracite in the American blast furnace.

The earliest attempts to use anthracite for smelting of iron was made in 1820 by the Lehigh Coal and Navigation Co., which erected a small experimental furnace near Mauch Chunk, in Carbon County, Pa. Mauch Chunk was modeled after the plant at Vizille on the border of France and Switzerland. The Vizille trial was conducted under the supervision of the most famous blast furnace men of Europe, Geynard and Robin, who in 1810 concluded that in furnaces operating on cold blast, use of anthracite in the burden is usually detrimental.

At Vizille, the maximum amount of anthracite which could be charged into the furnace was established at 40 pct. Beyond this level, the furnace began to display strong tendencies to hang. At approximately 50 pct of anthracite in the fuel burden, the Vizille furnace froze entirely. The results at Mauch Chunk confirmed those of Vizille. In May of 1826, W. McDowell, manager of the furnace, recommended to George Crane that further tests be temporarily discontinued, since the furnace was solidly frozen.

The furnace was dismantled and remained inactive until 1837 when a new series of anthracite ex-



A bloomery similar to the type operated by Col. Alexander Spotswood in Virginia.

periments was started, only to be discontinued in 1849. The failures, however, did not produce a general discouragement and resignation. In fact, they became a challenge to the intelligence and know-how of the blast furnace men who had to use anthracite to survive. They took up the challenge, and created the glorious era of anthracite iron.

Hot blast

During the early decades of the 19th century, the production capacities of blast furnaces were increasing continuously, though at a comparatively slow rate. While an average American furnace was making about 18 tons per week around the turn of the century, production increased to 21 tons by 1806, and 34 tons by 1827. The increase was achieved, however, by increasing the size and blast volume of the furnaces, and by improving the quality of the ore burden. The turn of the century was a milestone of the ironmaking processes since somewhere around this time the operators began to impose stricter requirements on the quality of their ores, the first steps being manual elimination of larger lumps of rock.

English iron masters watched with growing concern the rapidly diminishing reserves of wood for production of charcoal. Experiments with mineral coal as blast furnace fuel continued, therefore, without interruptions in all major iron works of the British Isles. The solution was nowhere near, however, when it occurred to the mind of the Scotch ironmaker, James Beaumont Neilson, in 1828, to reduce the consumption of fuel in the furnace by preheating the blast. In spite of the opposition of many contemporary physicists and chemists, the idea was tried in 1829 at the Clyde Iron Works and, to the surprise of all, it worked.

The first hot blast apparatus of Neilson was very simple. The stove consisted of a rectangular wrought iron heating chamber, about 4x2 ft, and 3 ft high. The chamber was set in brickwork which was also equipped with a fireplace underneath. The top-plate of the chamber was left exposed to the atmosphere. The cold blast entered the chamber immediately over the grate and passed out from the opposite end directly into the tuyere at a temperature of about 200°F.

Each tuyere of the furnace was equipped with one heating chamber. The effect of even this moderate heat was truly astonishing. The production rate of metal increased overnight by about 100 pct without increase of fuel. Neilson became the center of attention of the European ironmakers who pilgrimaged to the Scotch plant. Neilson's idea, however, contributed little to the change of the attitude of these blast furnace men as far as construction of the furnaces is concerned. Satisfied that his invention considerably improved the productive capacity of their furnaces, they kept building in the same traditional manner. Thus, typical furnaces built from 1820 to 1850 were 45 to 50 ft high with bosh diameters from 10 to 16 ft.

In the meantime, Neilson, realizing the shortcoming of his first blast heating apparatus, kept working on improving the heating chamber. The next step was a cylindrical vessel of cast iron, bottle shaped at each end, 2 ft 9 in. in diam and 6 ft long. The chamber was fixed in horizontal position over a fireplace and wholly enclosed in brick work to reduce the heat losses. The temperature of the blast was thus raised to 280°F.

Still unsatisfied, Neilson invented a new pipe heating chamber which raised the temperature to 600°F. The new apparatus consisted of an arrangement of cylindrical cast iron pipes 18 in. in diam, fixed horizontally and united by flanges. These pipes formed a continuous length of 100 ft and offered to the blast 240 sq ft of heating surface, which was more than four times that of the bottle-type heating chamber. The grate area on each of the heating chambers measured 28 sq ft. In this apparatus the actual hot blast was first produced. The pipe stoves of Neilson showed great disadvantages, of which the most important was breakage of pipes. This occurred usually in places of contact with carbonaceous fuel resting on the grate, which could not be avoided.

Arched pipes, introduced by Neilson as the next improvement, helped only to a limited degree. In order to prevent overheating, the arch pipes were elongated into syphon-shaped tubes and removed from the proximity of the grates. Thus the pipes conducting the blast were heated by hot combustion gases produced in the separate adjoining fireplace.

Still another improvement in the blast heating apparatus found its way to the Pennsylvania iron works of the 19th century. This one was called the box foot pipestove, and consisted of a series of cast iron boxes, each of which had two sockets on top. The heating pipes were syphon shaped and were set vertically in the respective sockets of two adjacent boxes. The blast passed up one leg of pipe and down the other, and so on through the entire system. These stoves were originally built at North Staffordshire, England, around 1848. They were subsequently listed as Neilson's box foot installations at several plants of the Lehigh Valley. All of these ovens were fired by solid fuel, preferably hard coal.

Another type of stove, called the oval oven, was developed by Josiah Smith in England around 1852. Smith inserted a straight length of main between the ends of each of the semi-circular mains of the conventional box stove, thus increasing the number

of the pairs of pipe from 24 to 33. This, according to data in the literature, was found to be such a great improvement over other blast stoves that another oval oven was erected in which the mains were further elongated to accommodate 36 pairs of pipe. The Parkfield furnaces near Wolverhampton and a dozen installations in nearby New Jersey and Pennsylvania were equipped with this type of blast heating apparatus.

Utilization of waste gas: 1832

The next improvement in the development of the blast furnace is rightly attributed to Fabre du Faur, who about 1832 obtained a patent on the application of the blast furnace waste gas to the preheating of the blast. Following the original experiments of du Faur in Germany, the idea was picked up by Perry at Ebbwvale, England, and found to be extremely economical. The work was described by Percy as follows:

> "At Blaina and Ebbwvale I saw the application of waste gas of the blast furnace carried out apparently to perfection. I have never witnessed any metallurgical operation with more pleasure than that of these hot blast stoves and I felt no small degree of commiseration for iron masters who still pursue the old plan of using solid fuel such as coal-slack however cheaply it may be obtained. Mr. Levich of the Blaina Iron Works assures me that the savings affected at his works by the application of waste gas of the blast furnaces to the heating of the hot blast and steam boilers is equivalent to 600 tons of coal a week".

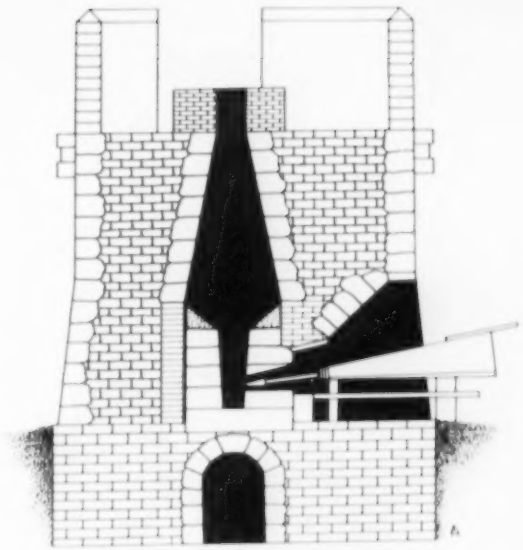
It was this type of hot blast stove which was installed between 1854 and 1856 on three coke furnaces of the Cambria Steam Hot Blast Coke Furnace Co. located at Johnstown Flat, in western Pa. The exact size of the Cambria blast stoves is not known, but it is significant that the plant was the first one in the U. S. to use one large steam blowing engine to provide blast simultaneously to three separate furnaces. It is also of interest to note that Kelly's converter process was put to test here between 1855 and 1860.

The Blaina hot blast stove was the forerunner of the Cowper stoves which eventually became the standard for the entire blast furnace industry. The gas heated blast stoves belong in fact to the period of coke furnaces. It was the coal heated blast apparatus of Neilson and Thomas which actually broke the spell of small charcoal furnaces and put the anthracite furnace in operation, thereby contributing considerably to the growth of the mightiest steel industry of the world.

The anthracite glory

The era of the American anthracite blast furnace was born in 1839 when David Thomas blew in the Pioneer furnace at Pottsville, Schuylkill County, Pa. Thomas was an Englishman who came to this country on the invitation of Mr. Erskin Hazard of the Lehigh Iron & Navigation Co. for the distinct purpose, clearly specified in his contract, of building initially one and then other anthracite furnaces for the company.

The Yniscedwin Iron Works in Wales, where Thomas worked as a young man, were on a deposit of anthracite coal, which was considered useless as far as operation of blast furnaces was concerned. Thomas thought frequently about using



Profile of an early American charcoal furnace.

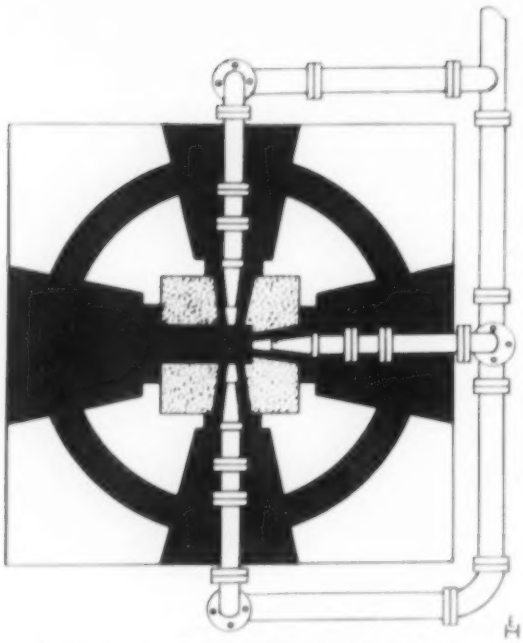
anthracite, and with the assistance of George Crane, the owner, proceeded to experiment with anthracite as partial fuel burden of his furnaces as early as 1820. The amount of anthracite was small and never exceeded 8 pct of the total fuel burden. According to Thomas, the furnace performed quite well under these conditions, but whenever something went wrong, the anthracite was blamed. Finally the furnace operators became prejudiced against the raw fuel and Thomas was forced to give it up. Thereafter, he repeated his experiments almost every year. In 1825, he built a separate experimental furnace for the test runs with anthracite. The furnace was 28 ft high and had a 9-ft-diam bosh. Thomas burdened this furnace with increasing amounts of anthracite, but to no avail. The furnace ran cold and the tests had to be discontinued. Thomas rebuilt the furnace in 1830. He extended the shaft to 45 ft height and widened the bosh to 11 ft diam. The experiments were much more successful than previously, but the consumption of coal was so high that they had to be abandoned.

About this time experiments were also being made with anthracite at Mauch Chunk, Pa., with just about the same success.

In 1829, while the experiments were going on on both sides of the Atlantic, Neilson of Glasgow, totally unaware of Thomas' problems, obtained a patent for his hot blast apparatus. Thomas and Crane immediately recognized that Neilson's discovery might be the key to the successful utilization of anthracite in the blast furnace. Consequently, Thomas traveled to the Clyde Iron Works in Scotland to see the operation. Thomas returned to Yniscedwin with Neilson's license, now thoroughly convinced that hot blast would solve his anthracite problem.

Together with a mechanic brought from the Clyde Iron Works, Thomas built the first blast stove for an anthracite furnace, which was blown in on Feb. 5, 1837. The operation of the furnace was a complete success and the furnace produced pig iron of good quality at a satisfactory rate.

In May of the same year, a Mr. Roberts from Philadelphia came to visit George Crane, saw the furnace in operation and reported this immediately to his uncle, Josiah White of the Lehigh Coal and Navi-



Tuyere arrangement of a 19th century anthracite furnace.

gation Co. White, who previously had participated in many of the experiments on the use of anthracite at Mauch Chunk, immediately grasped the significance of Thomas' furnace and dispatched Mr. Erskine Hazard, his friend and vice president, to evaluate the development in England and to bring to this country someone who could build and operate an identical furnace in Pennsylvania. Hazard arrived in England in November 1838, found the furnace in full operation, and began negotiations with George Crane.

Hazard convinced Crane that Thomas could be more valuable in the U. S. than in England. Together, they went to see Thomas. Crane and Hazard presented Thomas a realistic picture of the blast furnace problem in America, and urged Thomas to accept the call of the new nation. It was a difficult decision; years of peaceful and satisfying life in the rural surroundings of Wales, and countrywide acclaim as inventor of the successful anthracite blast furnace assured him everything for which he dared to hope. However, the new world contained a challenge which Thomas could not refuse.

Thomas comes to the U. S.

The contract was signed on Dec. 31, 1838, and the Thomas family sailed in May 1839. Thomas arrived in Allentown, Pa., and proceeded almost immediately with the plans for the future iron works. Surveys and plan drawings were completed about August 1, and the work was started on excavations for the foundation of the wheel pit, which was to be the raceway to the water wheels, and the canal for supplying the raw materials to the furnaces.

A few weeks later, foundations for the furnace itself were started. The furnace was to be 30 ft square at the base, having a 12 ft-bosh and a height of 45 ft. The masonry was laid by Isaac McHose, whose son Samuel was subsequently builder of almost all anthracite furnaces in the Lehigh Valley. The hot blast stove of the furnace consisted of four ovens of 12 arched pipes each. The pipes measured 5 in. ID, 1½ in. in thickness in the straight sections, and were 2 in. thick in the arches. The stoves were built on the ground and fired with coal. The joints on the pipes were made with liquid cast iron, the point of junction of the arch pipes being carefully sealed to prevent the iron from running into the bed pipes. After the joints were made they were covered with salt and sal-ammoniac water which rusted them perfectly tight. The stoves were capable of heating the blast to approximately 600°F.

The arrangement for filling the furnace consisted of a water balance-type elevator in which square boxes were attached to each end of a chain. The chain in turn was wound around a wheel with a brake. While water was filled into one of the boxes, the load in the other box was lifted to the top of the furnace. As soon as this happened the water box touched a trip mechanism which emptied the water from the box and the entire procedure would be repeated.

The first American blast furnace built exclusively for anthracite was blown by a breast wheel 12 ft in diam and 24 ft long. Water falling from a height of 8 ft furnished the power. On each side of the wheel were segments geared into pinions on double tracks driving two blowing cylinders. The cylinders were 5 ft in diam and had a 6-ft stroke.

The blast from the cylinders was subsequently conducted through an 18-in. cast iron pipe to the stoves and hence to the furnace. The flow of the blast was pulsating constantly and the strokes of the cylinders could be counted as well at the furnace as at the wheelhouse. The blowing cylinders were made out of necessity in the U. S., because cargo ships refused to take aboard the huge English machines weighing several tons each.

Merrick and Towne built the cylinders and subsequently received numerous other orders from the mushrooming anthracite industry. The original cylinders which were made in England, arrived a few months later and were used in various installations as late as 1905. After many delays, the No. 1 Crane Co. furnace was blown in at 5 pm on July 3, 1840.

Use of Anthracite Expands

The Catasauqua furnace was not the first American blast furnace to operate on anthracite, for after extensive experimental runs in 1838 and 1839, the Mauch Chunk furnace was reactivated as an anthracite furnace, David Thomas helping with advice. It was not the old Mauch Chunk furnace any more; the furnace had been rebuilt in 1837 at a site about half a mile closer to the village of Mauch Chunk. The stack of this furnace was 21½ ft high, 22 ft square at the base, and 5½ ft across the bosh. The hearth had a rectangular cross section measuring 14x16 in.

Blowing apparatus consisted of two cylinders 6 ft in diam actuated by a water turbine 14 ft in diam and equipped with 36 water buckets. Design of the hot blast apparatus is not described in the literature. It was built, however, according to a drawing provided by Thomas shortly after his arrival in the U. S. The furnace was blown in on August 27 and operated continuously until Sept. 10, 1839, when the run was discontinued because of difficulties in the hot blast apparatus.

The Pottsville furnace of the Crane Co. was the scene of a similar experiment in the same year. The results indicated, however, that the basic approach of Thomas, who also designed the Pottsville hot blast apparatus, was entirely sound. The operation at Pottsville was resumed in August 1840, about one

month after the blowing-in of the Crane Co. No. 1 furnace at Catasauqua, Pa.

The date is a memorable and important one because the Catasauqua anthracite blast furnace proved to be a milestone in the steel industry of the U. S. This furnace was the model for the development of the entire Lehigh Valley iron industry, which was later to give the national armies under Ulysses Grant the decisive advantage over the industrially undeveloped South.

To David Thomas must go much of the credit for this tremendous new development. By accepting a position with an unknown American organization, he gave the activities of the young and energetic people all he had—his knowhow and his love of the iron trade.

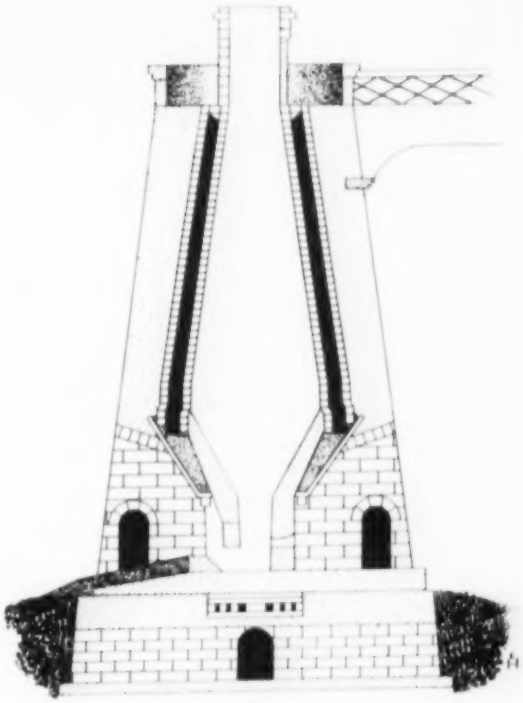
The first Thomas furnace remained on blast until it was ruined by the great flood of January 1841. In the period of the six months of operation the furnace produced 1080 tons of iron with the maximum weekly output of 52 tons. In the meantime, the ore mines had to be developed and transportation of raw materials provided. Ore mines were established in the immediate vicinity of the furnace with none of the hauls longer than 32 miles. The metallic mix was on the average 25 pct magnetite and 75 pct hematite. The No. 1 Catasauqua furnace was blown in again on May 18, 1841 and until its shutdown on Aug. 6, 1842, produced 3316 tons of pig iron.

Thomas, now famous, was highly respected throughout Pennsylvania. He was frequently consulted by enterprising men on various problems encountered in building anthracite blast furnaces. The first ones to follow in the footsteps of the Crane Co. were H. Post of Stanhope, N. J., Henry at Scranton, Firmstone at Glendane, Governor Porter at Harrisburg, and Dr. Echert at Reading. By 1846, there were 40 anthracite furnaces concentrated in clusters along the Lehigh, Schuylkill, Hudson, and Susquehanna Rivers.

Thomas was involved to a greater or lesser degree in almost every one of these installations. Usually it was only to advise and suggest. From time to time, however, he drew plans for the installations and as his time allowed, supervised construction himself. As far as could be determined, Thomas built a total of five furnaces for the Lehigh Crane Co. in the decade ending in 1850.

The anthracite iron masters were truly progressive. In 1847, a novel experiment was tried on Crane Co.'s No. 3 Furnace, by passing a strong electric current through the molten iron collected in the hearth. The thought behind this was to reduce the phosphorus content of the iron by electricity, which at that time was considered a cure for all ills. This experiment was terminated quickly when the current from a 100-cell battery caused only a severe electric shock to the blower foreman but not the slightest reduction of the phosphorus content of iron.

During 1849 and 1850 the Crane Company added two more furnaces to its famous plant. Furnaces 4 and 5 were identical and measured 18 ft in bosh diam and 45 ft in height. The blowing engines on both these furnaces were the largest made heretofore in the U. S. They had a 7-ft-diam cylinder, a 9-ft stroke and could blow under pressure up to 5½ psi, which was quite unusual in those times. After the first few months of operation it became evident that the furnaces were too short for the volume of available air (9500 cfm). Consequently, they were raised to 55 ft in 1852 and from then on, each produced on the average 300 tons of iron per week. Starting with 1857, the furnaces held the production records of all American anthracite blast furnaces, each averaging upwards of 10,000 tons of iron a year.



Profile view of a 19th century anthracite furnace.

Thomas left the Lehigh Crane Co. in 1854 and established his own iron and steel company with two blast furnaces at Hockendauqua, Lehigh County, Pa. The plant was located on the Lehigh River about one mile above the Crane Works. It was connected by a spur with the main line of the Lehigh Valley Railroad. The plant was only four miles from Allentown, Pa., and was managed by David Thomas and his son Samuel; it was regarded as the example of an efficiently operated plant.

Both Thomas Co. furnaces were 18 ft in bosh diam and 60 ft high. They had one common blowing machine consisting of two cylinders blowing under pressure of 8½ psi. Pig iron production of these two furnaces kept increasing gradually from 17,446 tons in 1856 to approximately 23,000 tons in 1859. Not much is known about the fate of the Thomas furnaces, except that they were abandoned like other anthracite furnaces early in the 20th century.

Production of anthracite pig iron in America increased rapidly from approximately 22,000 tons in 1842 to 393,000 in 1856, of which 306,000 tons were produced in Pennsylvania. The mushrooming iron industry contributed considerably to the changes in the living standard and social structure pattern of the young Nation. The heavily populated areas of the Eastern basin represented strong drawing points for the European immigrants to the U. S., who in turn added skill and perseverance to the industrial impetus of the growing world power.

The final part of *Blast Furnace U. S. A.* will appear in a forthcoming issue of JOURNAL OF METALS.

The Cleveland Conference

High-Temperature Materials

A Technical Summary

"The development of new high-temperature alloys which are based on the high melting point refractory metals will be expensive and there will be many difficult problems to solve, but the stakes are high."—R. H. Thielemann, Stanford Research Institute.

MATERIALS attractive for use at 1500°F in aircraft, missiles, and nuclear reactors were the topic of discussion at the Cleveland High-Temperature Materials Conference, held in April of this year under the sponsorship of the IMD High-Temperature Alloys Committee.

A special feature of this meeting were survey papers prepared to give a general outline of work being done in the various fields that were discussed. Following are extracts from several of these papers, with the accent placed on the present and future prospects in this rapidly expanding field.

Observations of SAP and Present Theories for Its Remarkable High Temperature Strength

Fritz Lenel, Rensselaer Polytechnic Institute.

Another aspect of dispersion strengthening is the role of some of the strengthening effects which have been proposed as being reponsible for the properties of SAP-type materials: strengthening and immobilization of grain boundaries by the second phase, and the storage of strain energy during extrusion of the material. I do not want to imply that, on theoretical grounds, these effects can be cleanly separated from each other or from the principal room temperature effect of the dispersed particles—the prevention of slip. Nevertheless, we should like to know as much as possible about the contributions of each of these effects to the properties of the material to help better overall understanding of the dispersion strengthening mechanism.

The effects of cold working upon the properties of aluminum powder extrusions made from fine flake powders was studied by the Swiss investigation. They found that the extrusions became only a little stronger during cold working and that heating the cold worked extrusions to 932°F lowers the strength only to the as-extruded value and does not cause recrystallization. Grant made similar observation on the hardness of SAP extrusions which led him to postulate that strain energy is stored in the lattice during extrusion. We made similar experiments on extrusions from somewhat coarser aluminum powders, an atomized powder R-3M with an average particle diameter of 3μ and a flake powder, MD 2100, with an average flake thickness of 0.8μ. The extrusions were in the form of wires 0.088 in. diam so that they could be readily cold worked by drawing them through drawing dies, until the cross section of the wire was reduced 84 pct to a diameter of 0.035 in. The cold worked wires were heated to a series of temperatures from 200° to 1050°F and the tensile strength of the wires was determined. The results are shown in Fig. 1. Both types of extrusions increase in strength during the severe cold working, the percentage increase for the more finely dispersed MD 2100 powder extrusion being less than for the coarser R-3M powder extrusion. Even the latter is strengthened much less than annealed commercially pure aluminum would be. The sudden decrease in strength of the cold worked extrusions after heating to a well defined narrow temperature range is, of course, typical for cold worked materials. Examination by X-ray diffraction and by metallography clearly show that the extrusions actually recrystallize. The resulting grainstructure of the MD 2100 powder extrusions is very coarse.

The tensile strength of the recrystallized wires is lower than that of the as extruded wires, but both recrystallized wires are much stronger than annealed commercially pure aluminum. As expected, the strength level for the finer dispersion from MD 2100 powder is higher than for the coarser dispersion from R-3M powder. Since the recrystallized material contains only a few large-angle grain boundaries and should be essentially strainfree, the higher strength of the recrystallized materials must be due to the effect of the dispersion upon prevention of slip. This investigation is only in its beginning. We shall try to recrystallize materials with finer dispersions of aluminum oxide. In addition to room temperature properties, we expect to measure elevated temperature strength both short time and in stress rupture. The structure of the recrystallized material is to be examined under the electron microscope for possible effects of cold working and recrystallization upon the shape and distribution of the dispersed phase. This work should contribute to our understanding of the dispersion strengthening mechanism.

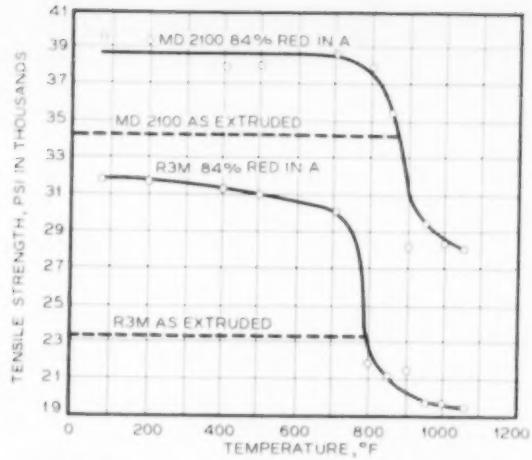


Fig. 1—Tensile strength of wire drawn from aluminum powder extrusion as a function of the annealing temperature. R3M is an atomised powder with an average particle diameter of 3μ and MD 2100 is a flake powder with an average flake thickness of 0.8μ.

Status and Future of Cobalt-Base Alloys

Glenn Fritzlen, Haynes Stellite Co.

Two wrought cobalt-base alloys* have been applied to solve specific high temperature problems within the last decade. One of these alloys having 0.40 pct C has been used principally in the forged, solution-treated, and aged condition for rotating parts of turbosuperchargers and turbojets, while a second was originally designed to be used in the solution heat-treated condition as a readily fabricated sheet material for afterburner components.

Since many of the recent engineering developmental projects require increasing amounts of high strength sheet, experiments have been conducted recently to explore whether strain-hardening and aging of the Co-Cr-W-Ni-low carbon alloy could increase high temperature yield and rupture strengths. Fig. 2 shows room-temperature tensile test results from specimens which were reduced up to 20 pct in thickness by cold rolling operations. A second series of specimens was given similar amounts of reduction and then was subsequently aged for 4 hr at 1100°F. One may see the marked effect of cold work on increasing yield strength and lowering elongation values; however, even at 20 pct reduction, there was 27 pct elongation retained in the sheet which might indicate sufficient formability. It may be feasible to process sheet material which is in a cold-worked temper corresponding to the range of 10 to 20 pct reduction and then to age the sheet prior to exposure to heat in service. Table I gives some short time tensile properties obtained from sheet specimens in the solution-treated, cold-reduced, and cold-reduced plus aged condition. Marked gains in strength may be derived from the cold-reduced tempers and, also, it appears possible to gain additional benefits if the proper aging treatments are administered to previously cold-reduced material.

The yield strength value divided by the density gives an index which is used as a quide by materials engineers, particularly, in the selection of materials for design of aerial vehicles. A goal of 800,000 has been established for this index to reach at 1200°F. A plot of yield divided by density indices is shown in Fig. 3. Although the target of 800,000 was not attained, improvements in the index were achieved. It is believed that moderate amounts of cold work from about 5 to 20 pct followed by the relatively lower aging temperature (1100°F) will be beneficial in improving high temperature strength in the service temperature range where transgranular failures would be expected. Recent electron micrographic and optical metallographic studies have revealed that relatively coarse M_6C particles are formed after 16 hr at 1380°F, while the lower temperature treatment of 4 hr at 1100°F did not produce the same abundance or as discrete particles. The larger particles produced abundantly by cold-reduction followed by aging for 16 hr at 1380°F may be generally more effective for short time improvements in strength and life at temperatures where intergranular fracture would be expected.

Stress-rupture tests from 1500° to 2000°F were conducted on bar stock specimens which were cold worked and aged. Significant increases in rupture lives up to 2000°F were obtained with 10 pct cold reduction followed by aging for 16 hr at 1380°F. The greater amounts of cold reduction did not yield as

* Allegehany-Ludium S-816 Alloy (40 pct Co-20 pct Cr-20 pct Ni-4 pct W-4 pct Mo-4 pct Cb-0.4 pct C). Haynes Alloy No. 25 (Co-20 pct Cr-15 pct W-10 pct Ni-0.10 pct C).

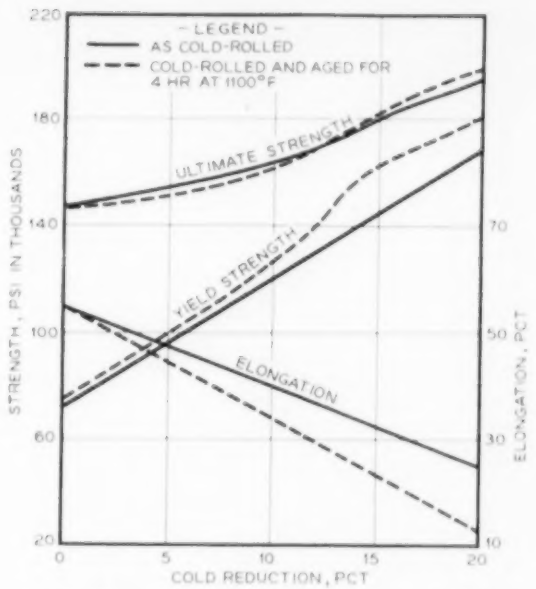


Fig. 2—Effects of cold reduction on Co-20 pct Cr-15 pct W-10 pct Ni-0.10 pct C alloy sheet, (Haynes alloy No. 25).

Table I. Effects of Cold Reduction and Aging on Co-Cr-W-Ni Alloy Sheet Short Time Tensile Properties

Test Temperature, °F	Ultimate Tensile Strength, Psi	Yield Strength, 0.2 Pct Offset, Psi	Elongation in 2 In., Pct	Index of Yield Strength/ Density in 1,000's
Solution Heat-Treated Condition, No Cold Reduction				
70	155,000	67,000	55	201
As-Rolled Condition, Tested After 10 Pct Cold Reduction				
70	165,000	120,000	43	360
1200	127,500	87,000	18.5	262
1500	94,000	67,000	13	200
1800	41,000	26,000	16.5	79
Aged at 1380°F For 16 Hr, After 35 Pct Cold Reduction				
1200	160,000	107,500	8.5	322
Aged at 1380°F For 16 Hr, After 40 Pct Cold Reduction				
1200	150,000	106,500	7.5	320
Aged at 1380°F For 16 Hr, After 45 Pct Cold Reduction				
1200	147,000	116,500	6.5	350

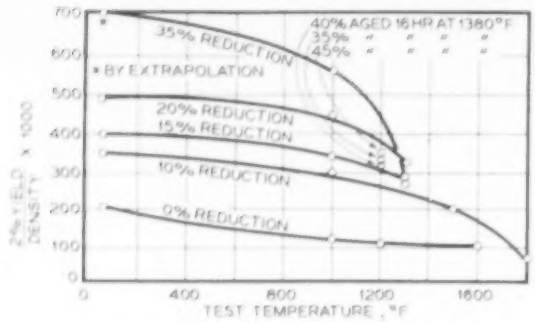


Fig. 3—Two pct yield/density of cold rolled Haynes alloy No. 25, tensile tested at various temperatures.

large an increase in life. The criticality of the treatments to be utilized is evident, but there seems to be a practical range within which to operate.

From this brief survey, one may see the potential value of cold-work followed by selected aging treatments to provide stronger high temperature materials for stressed skins and structural members, as well as other highly loaded components of military and commercial apparatus.

Cermets and Bonded Hard Metals

John T. Norton, Massachusetts Institute of Technology.

Cermets have found application in two important areas. The first of these involves materials for various kinds of tools, and in this area the cemented carbides have achieved an unqualified success, outstripping all competitors. Producible reliably with a wide range of properties, we see them in a whole gamut of applications such as fine finishing and heavy roughing tools, wire drawing dies, rock drills, and cold heading tools all serving faithfully in the process of persuading a reluctant solid into useful shapes.

The second area of application of cermets has involved materials from which to make structural parts for high temperature service. It was only natural to expect the kind of material which had been successful in the tool field would be a possible answer if the components were modified to meet the new conditions. The important new criterion of course was increased resistance to oxidation. A host of combinations of hard phase and binder metal have been studied and presently the metal-bonded titanium carbide group of alloys stands well in front. Part of the pre-eminence that this group enjoys is due to something inherent in the nature of the material itself but certainly a considerable portion is due to the magnitude and intensity of the attention it has received. In any event, if we look back over the progress of the last five years, I believe we must admit that as far as engineering development is concerned we seem to have reached a sort of plateau and that our best efforts have not been able to produce a significant break-through.

It seems to me that the properties of an aggregate structure, strength and hardness, and their variations with temperature and with rate of loading, must be uniquely related to the properties of the components themselves and to the microstructure. By microstructure, I mean the shape, size, and size distribution of the hard phase as well as manner in which the binder phase distributes itself around the hard particles. If this is correct then in order to develop the idea we must have fairly complete answers to three questions:

1) *What are the mechanical properties of the components?*

As to the properties of the hard phase, we are in a very poor position. Only very approximate estimates are available as to the overall mechanical properties of these substances. In order to obtain the required information it is imperative that pure single crystals be prepared of such a size that the properties can be measured and their variation with temperature and crystallographic direction be demonstrated.

2) *What is the ideal microstructure consistent with the properties of the components?*

One might conclude that the ideal structure would be one of small grains of the hard phase in the form of polyhedra regularly packed with very thin layers of binder metal between. Or alternatively, an array of spherical particles of appropriate size distribution to give dense packing but again separated by thin films of binder. Possibly a compromise is required.

And of course one should not overlook the possibilities of a fibrous texture in which long rod like crystals of the hard phase are embedded in the metal matrix.

3) *What means do we have available for obtaining the ideal microstructure in the finished part?*

Assuming that the cermet is made by powder metallurgy techniques, the sintering process itself offers little in the way of external control. The time and temperature of sintering cannot be varied over very wide ranges and the variations in the atmosphere are limited. This means that the significant controls must be inherent in what goes into the furnace. In short, to obtain the intelligent answers, the whole problem of sintering in the presence of a liquid phase must be studied in much more detail than it has in the past.

Before considering the details of the program, it is necessary to take a very good look at the goals to be won. Such a fundamental program of learning to understand, applied to the metal-bonded hard metals would, I am confident, result in materials significantly better than those available today. What we must decide is whether this rather formidable effort is not directed toward too limited an objective. Are the cermets in the sense that we consider them today already out of date? I feel very strongly that the high-temperature structural materials of the future will involve some of the members of a group consisting of the oxides, carbides, nitrides, borides, aluminides, silicides, germanides, and possibly stannides of the transition metals and I believe that we have no other alternative but to press on to a better and more intelligent understanding of these materials.

We have concentrated on the problem of making hardware and as a result we have neither the hardware we want nor the understanding. If we now concentrate on the understanding, I believe a superior brand of hardware will be an automatic consequence.

Chromium-Base Alloys for High-Temperature Applications

R. W. Fountain and M. Korchynsky, Haynes Stellite Co.

On the surface, at least, chromium possesses many of the properties generally considered to be important for high-temperature applications.

In view of these properties, considerable effort has been expanded to design chromium alloys for high-temperature service. Most of the information on chromium alloys relates to their resistance to deformation at elevated temperatures, while only recently has attention been given to the physical properties.

The most serious problem which has prevented the utilization of chromium-base alloys is their brittle behavior at room temperature, which is shown not only in the highly creep-resistant alloys but also by the less strong alloys and chromium metal itself, even when in relatively pure form.

Experience gained from studies on the plastic behavior of other transition elements indicated that the room temperature ductility of these body-centered-cubic or hexagonal-close-packed metals can be tremendously improved by reducing the amount of impurities below a certain critical level. It appeared the same should be possible for chromium, and therefore much work has been concerned with the preparation of high-purity chromium and determination of its properties.

• Investigators at the Bureau of Mines, who studied the mechanical properties of electropolished wrought chromium wire prepared from hydrogen-reduced electrolytic chromium, obtained an elongation of 25 pct with an ultimate strength of 70,000 psi, using a low strain rate.

• Tensile properties of fully recrystallized chromium were determined at temperatures up to 800°C, and virtually no ductility was observed below 350°C. Above this temperature, the ductility increased abruptly, reaching a maximum of approximately 50 pct elongation at 400°C. At 500°C the elongation dropped to about 30 pct but subsequently increased with a further increase in test temperature. The ultimate tensile strength tended to increase with temperature, reaching a maximum of 40,000 psi at 500°C. These data indicate that structural use of pure chromium metal in very high-temperature applications appears unlikely.

• Investigators at the Bureau of Mines found that arc-melted chromium which could not be worked below 500°C could, nevertheless, be cold-worked up to 60 pct by intermittent refrigeration to liquid air temperature (−190°C) after each 5 pct reduction. Suggestions made to explain this phenomenon postulated the existence of a low-temperature, ductile modification of chromium.

• It has been found that various physical properties of chromium show, with changes in temperature, anomalies which have not been satisfactorily explained. The most striking of these are the discontinuous changes which occur at approximately 37°C, in Young's modulus, internal friction, resistivity, thermoelectric power, and coefficient of thermal expansion. These changes are attributed to the properties of chromium itself and apparently are not caused, as suspected earlier, by the presence of impurities, particularly Cr_2O_3. There are no indications of an allotropic transformation of chromium at the temperatures at which the abnormal behavior takes place, and the lattice parameter shows only a slight break. The difference between the variation of the lattice constant with temperature and the linear expansion data suggests, however, that there is a deviation from the ideal body-centered-cubic arrangement on passing through the transition, corresponding to a denser packing above the transition than below.

• Careful measurements revealed that the lattice component of the thermal conductivity of chromium

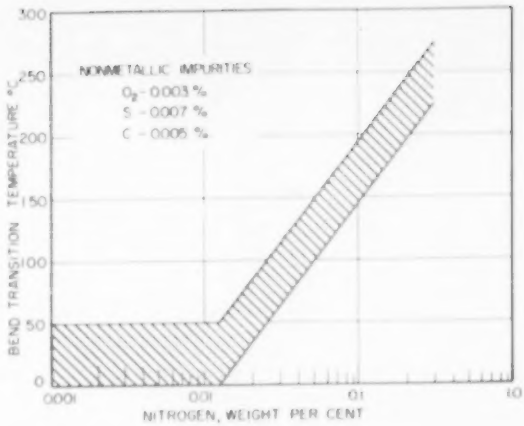


Fig. 4—Effect of nitrogen content on the bend transition temperature of hydrogen reduced electrolytic chromium (W. H. Smith and A. U. Seybolt).

is surprisingly high, accounting for about one third of the total thermal conductivity. In this respect, chromium differs from all other transition metals, a similar behavior being observed only in beryllium. Chromium also exhibits, unlike other transition elements, a definite antiferromagnetic behavior, the Néel temperature being about 200°C.

No explanation of these phenomena has been offered as yet, and there is no evidence as to whether there is an interrelation between the effects of temperature on the physical and the mechanical properties of chromium.

Later investigations have brought about an almost complete reversal of opinion on the embrittling role of oxygen. It has been found that up to 0.34 pct of this element does not have an appreciable effect on the brittle to ductile transition of as-cast chromium. As the transition temperature of chromium appeared to be insensitive to large variation in oxygen content, investigations appear to indicate that nitrogen is the most detrimental impurity, the critical nitrogen content being as low as 0.002 to 0.003 pct or even less, as shown in Fig 4. Nitrogen in solid solution is more detrimental to ductility than when precipitated as chromium nitride.

Attempts to minimize the harmful effects of these nonmetallic impurities by additions of scavenging elements, such as aluminum, tantalum, titanium, or zirconium, were quite disappointing. Partial success was achieved only by cerium addition.

The detrimental effect of metallic alloying additions on the ductility tends to pass through a maximum, and at still higher alloy contents an appreciable degree of ductility can be attained. Among the high-purity chromium alloys investigated, the Cr-Fe, Cr-Ni, and Cr-Co systems exhibit a significant range of fabricable alloy compositions. To maintain some room temperature tensile ductility, however, the limiting chromium content appears to be about 65 pct for the Cr-Fe, 60 pct for the Cr-Ni, and only 40 pct for the Cr-Co systems. In addition to the effect of composition, the tensile ductility of these alloys depends on the amount of hot-working.

. . . The successful solution of the chromium purification problem, however, has not fully substantiated the original expectations that higher purity would result in adequate ductility of chromium metal and, consequently, of chromium-base alloys.

The tensile ductility of chromium has been remarkably improved and the temperature of transition from ductile to brittle fracture substantially lowered. These improvements, however, have been determined only under certain testing conditions and on relatively small samples. The overall ductility of the purest chromium available still remains below the level generally required for engineering purposes. In the field of chromium-rich alloys, the effect of enhanced purity on the room temperature ductility and hot-workability is even less marked. It remains to be seen whether the ductility of these materials can be improved by processing technology.

It must be realized that this rather dim picture is based on the results of relatively few studies, and additional research is needed to evaluate more completely the effect of nitrogen, which is considered to be the most harmful impurity. Furthermore, the hypothesis that the critical impurity level in chromium is below that obtained by the present purification methods remains strictly speculative.

The systematic study of the effect of metallic additions on the transition temperature began only recently. It has been observed that there may be a relationship between the transition temperature of binary chromium-rich alloys and the position of the alloying element within the periodic system. Attempts are being made to correlate the degree of change in the transition temperature with the electronic structure of the solute atoms, and this approach might provide a rational basis for the design of alloys.

Effects of Vacuum Melting Upon Gas Content and Metal Structure of Cast Nickel Base Alloys

C. M. Hammond and R. A. Flinn, University of Michigan.

Vacuum melting, or more generally the variation of pressure and melt atmosphere during melting, refining, and casting, can have effects on control of gas content, and control of minor elements (normally nongaseous). These will be considered in turn.

I. Control of Gas Content

A) *Reactions between melt and atmosphere (H_2, N_2, O_2):* In the melting and refining of metals for elevated temperature service, the gases of major interest are hydrogen, nitrogen, oxygen, and carbon monoxide. The so-called simple gases, hydrogen and nitrogen, follow the application of Henry's law to the law of mass action. In general cases, if:

$$G_n = nG$$

where G = gas and n = the number of atoms per gas molecule, then:

$$\frac{(\text{activity } G)^n}{\text{activity } G_n} = K.$$

In the specific case of diatomic gases such as hydrogen and nitrogen, this becomes the familiar Sievert's law if we assume Henry's law holds, i.e., that activity is proportional to weight percent and pressure.

Then:

$$\text{pct H} = K\sqrt{\text{pH}_2} \qquad [1]$$

At this point it is important to point out that while the reduction in pressure over the melt lowers the gas content, the volume of porosity in ingot or casting caused by gas evolution upon solidification may not be reduced.

Hydrogen and nitrogen analyses may also be lowered by the CO boil discussed in the next section. Reactions with water vapor are important but have been thoroughly discussed in the past.

B) *Carbon deoxidation:* If hydrogen and nitrogen are reduced, the evolution of CO upon freezing becomes the principal source of porosity under normal atmospheres in iron and nickel-base alloys. To prevent this reaction, powerful deoxidants are added to reduce the dissolved oxygen and to act as buffers during solidification to prevent CO formation. This practice leads, of course, to the familiar spinel, corundum, and glassy inclusions. This effect is probably more important in bearing steels than in high temperature alloys.

Under reduced pressure, the reaction $C + O \rightleftharpoons CO$ (g) proceeds to the right far beyond the conditions for 1 atm. In fact, the evolution of CO from the liquid can be extremely violent if the pressure is reduced too rapidly. The CO bubbles also provide an interface for diffusion of H_2 and N_2 from the melt and thereby tend to sweep out the gases.

II. Minor Elements

A) *Removal of trace elements by distillation:* It has long been known that elements like manganese and zinc are removed rather easily at low pressures and steelmaking temperatures. It has been rather natural to ascribe some of the improvements in mechanical properties to these changes in trace elements.

In general, data are available for the vapor pressures of pure elements in equations of the form:

$$\log p = \frac{-\Delta H}{4.575\,T} + B$$

where p is the vapor pressure, ΔH is the molar heat of vaporization, and B is a characteristic of the substance.

However, we are usually interested in traces of elements dissolved in nickel or iron. In these cases we can, at present, only make rough approximations of the escaping tendency by assuming that the partial pressure of an element will be proportional to its mol fraction times the pressure calculated from the equation above. Deviations from the ideal condition can be severe and therefore, determinations of activities are needed for many important systems.

B) *Reduction of trace elements:* The reduction of trace elements from crucible materials has not received a proportionate share of attention until recently. The reduction of zirconium from zirconia offers an interesting example in the case of the nickel and iron-base alloys.

For iron-base alloys, we know the equilibrium constant for $ZrO_2(s) + 2C_{(in\ Fe\ or\ Ni)} \rightarrow \underline{Zr}_{(in\ Fe\ or\ Ni)} + 2CO(g)$.

For nickel-base alloys one approach is to use data for liquid iron, assuming the activity of the carbon and zirconium in the nickel alloy will resemble that for iron. This gives p_{CO} of the order of 10^{-6} atm.

In other words, at refining temperatures, if the pressure of CO is reduced to 10^{-6} atm at equilibrium, appreciable zirconium should be found in the melt. The reaction is therefore favored by the low pressures of the vacuum furnace.

The Los Angeles Reactive Metals Conference

A Technical Summary

"I submit that in our country, under a social and political system granting free rein to the initiative and creativeness of its people, many hidden mineral resources that otherwise would have lain fallow have been developed into productive enterprises to the benefit of our national industrial economy."—James Boyd, formerly Director, U. S. Bureau of Mines, now Vice-President, Kennecott Copper Corp., speaking at the Conference dinner.

"METALS for Today and Tomorrow" was the theme of the Regional Reactive Metals Conference, held in Los Angeles on May 28 and 29, and one could not help but acquire the enthusiasm that both the speakers and audience held for their subject. The feeling that big things either are happening or soon will happen seems to be generic among those who work with these 18 odd metals, with, of course, the major hopes being pinned on propulsion and nuclear reactor problems.

Although many of the projects reported will ultimately be published as completed research, the pace at which advances are being made in this field demands that a periodic summary be presented, showing the new directions that reactive metals research is taking.

Hafnium, zirconium, and titanium

Hafnium—Its Metallurgy, Properties, and Applications—*E. T. Hayes, U. S. Bureau of Mines.*

Hafnium is the last of the triad Ti-Zr-Hf to be commercially exploited. Its remarkable chemical similarity to zirconium, lean ores, and refractory nature combine to make the cost on the order of $100 per lb. The metal must be melted and worked using the same precautions observed by the titanium and zirconium industry to avoid contamination. The high atomic weight and elevated α-to-β transformation temperature suggest that the amount of alloying elements that can be dissolved in hafnium will be quite limited. The metal has only one use to date; its high absorption section for thermal neutrons makes it an excellent regulator for certain types of atomic power reactors.

Zirconium—*Haruo Kato, U. S. Bureau of Mines.*

Zirconium is of interest for nuclear energy applications because of its high melting point, excellent corrosion resistance, and suitable mechanical and physical properties. Current commercial production of the metal by the Kroll process exceeds 7 million lb annually. Like titanium, zirconium is very susceptible to the absorption of interstitial elements at high temperatures; however, it can be readily fabricated by casting, forging, rolling, and welding.

Porosity in Formed Titanium—*H. R. Ogden, R. A. Wood, D. N. Williams, and R. I. Jaffee, Battelle Memorial Institute.*

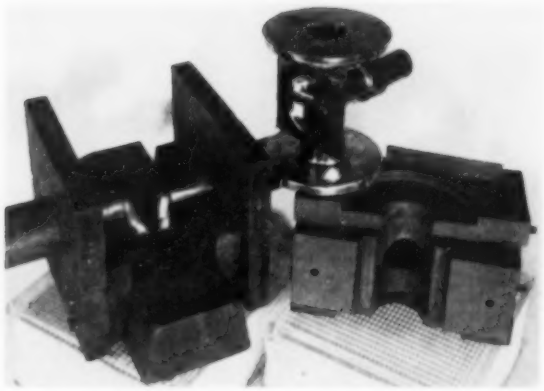
Surface pitting and internal voids have been found in formed titanium parts and in laboratory bend and tensile samples. These defects have been named strain-induced porosity because they only form in areas which have been highly strained, generally under triaxial stress conditions. Porosity increases with increasing strain and increasing temperature, being most severe in the temperature range of 300° to 700°F, at strains beyond the inherent uniform elongation of the material. Increasing strain rate decreases porosity while the presence of a notch promotes the formation of porosity in the triaxially stressed area near the root of the notch. Voids are generally located in grain boundaries, α-β interfaces, or interfaces between impurity phases and the matrix.

The Ti-7 Al-3 Mo Alloy: A Review of Its Development and Properties—*F. A. Crossley, Armour Research Foundation.*

The Ti-7 pct Al-3 pct Mo alloy has excellent combinations of strength and ductility in both Widmanstaetten and equiaxed microstructural conditions. The ductility of material with fine Widmanstaetten structure can be improved by solution treating at temperatures above 1500°F. Solution treating equiaxed material at 1800°F for 1 hr and water quenching and aging at 1000°F for 2 hr generally results in strength of approximately 200,000 psi and ductility of 5 to 10 pct elongation and 10 to 25 pct reduction in area. The upper temperature limit for application of material heat treated to high strength needs to be determined. The possibility that mate-

Titanium castings

A. H. ROBERSON, *Bureau of Mines*. Laminated machined graphite for titanium casting. These molds may be reused many times. In the event of breakage only the damaged part requires replacement. This feature extends mold life and lowers mold cost to a competitive level. This mold assembly has been used repeatedly without major repairs. The entire assembly consists of 18 segments.



. . . Diaphragm valve body and machined graphite mold. Laminating the mold makes it possible to transpose draft from the mold-casting interfaces to tapered wedge blocks. This allows greater draft to be used and at the same time reduces mold breakage. A taper of at least 7° is recommended for easy removal of the wedges. .

rial heat treated to high strength can be overaged to achieve stability without too great sacrifice of strength should be investigated.

Fabrication of reactive metals

The Vacuum Melting of Reactive Metals in Arc Furnaces—*S. M. Shelton, Oregon Metallurgical Corp.*

The reactivity of reactive metals is not the only problem in producing them as sound ingots or cast shapes. Consumable arc melting accomplishes some refining, but also accentuates some forms of segregation common to a degree in almost any type of melting. Techniques for improved soundness and homogeneity are still in the development stage, but show considerable promise. Inevitably, vacuum arc melting is establishing a field for itself; an extremely important field in metallurgical treatment, it is opening up new concepts and methods for producing substantial and consistent mechanical properties.

Extrusion and Forging of Reactive Metals—*G. A. Moudry, Harvey Machine Co.*

Titanium alloys have been successfully extruded and forged, and have found a place in the design manuals of the aircraft industry. Zirconium has just recently entered the extrusion and forging field; and the difficulties encountered during fabrication have been found to be similar to titanium, and, like titanium, much different from better known metals. Precautions must be taken to ensure a usable section having the required dimensional configuration and the necessary metallurgical structure and properties.

Welding Reactive Metals—*Bruce Baird, North American Aviation.*

Titanium, zirconium, and molybdenum are the three reactive metals on which the most welding development work has been done. The materials can be both resistance and fusion welded, and three different fusion welding processes can be used.

Application of Powder Metallurgy in Nuclear Engineering—*H. H. Hausner, Penn-Texas Corp.*

The four new metals to be used in nuclear power reactors are beryllium as a moderator, uranium and thorium for fuel, either in the form of pure metals, as alloys, or as cermets, and zirconium as a material of construction, especially for fuel cladding. The powders of these four metals are to be used usually with a high degree of purity. Greatest care has to be observed in handling of these metals in powder form, and they are to be treated in an entirely different manner than copper, iron, nickel, and other *ordinary* powders. There is a definite place for powder metallurgy in nuclear engineering, and the indications are that powder metallurgy will play an important, if not decisive, role in the development of materials and components for nuclear power reactors.

The alkali metals and thorium

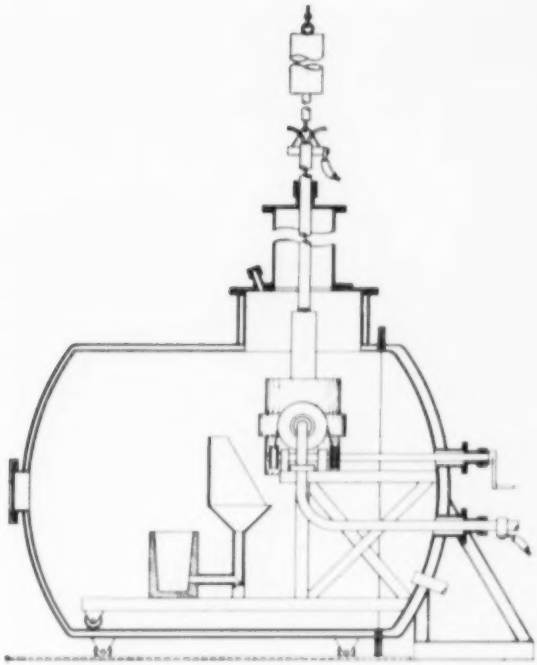
The Alkali Metals: Present and Future—*Marshall Sittig, The American Lithium Institute Inc.*

The alkali metals are booming in importance. New uses for sodium in metal reduction processes, new uses for lithium in making synthetic rubber, new availability of rubidium and cesium salts in tonnage quantities are all making news. Market trends for the various alkali metals are all on the upswing. Available in pure form in increasing volume, they offer sound applications today and wonderful possibilities for tomorrow.

Thorium in Atomic Energy—*C. C. Woolsey, Jr.*

Thorium is a source of nuclear fuel, not a fuel in itself. The three primary nuclear fuel materials are uranium-235, uranium-233, and plutonium-239. The first of these fissionable fuels occurs naturally, the other two being produced by neutron bombardment of natural materials. Such bombardment of thorium, and subsequent radioactive decay of intermediate products, results in production of the fuel material, uranium-233. The nature of the Th-U-233 system is such that, with a properly designed reactor, one can achieve breeding; that is, the reproduction of an atom of uranium-233 for each one fissioned. The Th-U-233 fuel cycle provides the highest neutron production ratio and hence is the system most likely to allow breeding in a thermal reactor.

. . . Integrally cast ¼ in. titanium chain made in a graphite mold. This illustrates the complicated shapes and excellent detail possible when properly designed molds are used . .



. . . Consumable-electrode casting furnace developed by the Bureau of Mines at Albany, Oregon. The furnace operates under vacuum and utilizes a tilting water-cooled copper crucible capable of pouring about 100 lb of metal.

Commercial Methods of Processing Lithium Ores—*L. Cohen and E. Berthold, American Potash and Chemical Corp.*

• Lithium Corp of America recovers lithium from spodumene by roasting and treating the roasted ore with acid. The lithium sulfate so formed is extracted with water and converted to lithium carbonate with soda ash. Lithium hydroxide can then be made from the carbonate by treatment with lime.

• Foote Mineral Co. mines and concentrates spodumene by flotation. The concentrates are roasted with an excess of limestone, and the resulting clinker is water leached to extract lithium hydroxide. The lithium hydroxide is recovered by evaporating leach liquor to produce solid lithium hydroxide monohydrate.

• American Lithium Chemicals imports lepidolite from Africa. The lepidolite is roasted with limestone and the calcine is leached with water. The process is roughly the same as the one used by Foote except that a lower roasting temperature is used. The recovery of lithium hydroxide is also complicated by the presence of various nonlithium alkalis also extracted from the lepidolite.

High melting metals

The Primary Fabrication of Arc Cast Molybdenum and its Alloys—*A. Timmons, Climax Molybdenum Co.*

Arc cast molybdenum and its alloys are produced in the form of cylindrical castings weighing 800 to 1000 lb. Unalloyed arc cast molybdenum, reduced less than 50 pct requires the minimum temperature of 2400°F for recrystallization, and some of the semi-commercial alloys require temperatures in excess of 2800°F for recrystallization. Furnaces in commercial forge shops and steel rolling mills are usually limited to temperatures near 2300°F, so true hot working is precluded.

Extrusion at temperatures near 2300°F has proven to be the most successful operation for accomplishing the initial (cold) breakdown of the cast metal. Extruded sections after annealing to produce complete recrystallization can be wrought successfully on hammer, press or rolling mill to produce desired semi-finished products.

True hot working has been accomplished in a single extrusion experiment in which a cast ingot was extruded at 3000°F. The extrusion exhibited the hardness of annealed molybdenum and transverse properties of the extrusion were markedly superior to the properties of extrusions produced at 2300°F.

The Metallurgy of Vanadium—*W. Rostoker, Armour Research Foundation.*

Future uses of vanadium and its alloys will be based on providing some optimum compromise of two or more mechanical and/or physical properties. At present, there are at least three possible applications for vanadium alloys:

1) Weldable, formable, high hot strength sheet alloy for airframe service at temperatures up to 1200°F.

2) Fuel element cladding for fast reactors where interdiffusion, hot strength, and thermal conductivity are considerations.

3) As a diffusion barrier between titanium and steel in the manufacture of clads between these two metals.

Physical and Mechanical Properties of Columbium—*J. H. Bechtold, Westinghouse Electric Corp.*

The demand for improved structural materials for use at high temperatures has resulted in considerable research on the refractory metals. Because of its low density, high melting point, good ductility, and promising elevated temperature strength, columbium is particularly attractive, particularly when compared with those of the other refractory metals in groups 5A and 6A of the periodic table.

Swedish Oxygen Steelmaking

In Sweden a rotating oxygen converter has been developed to produce open-hearth quality steel from high-phosphorus hot metal. Previously, the basic bessemer or thomas process with its quality disadvantages was used. The new method, called the Kal-Do process, is now to be installed in a second Swedish plant.

by Folke Johansson

PRACTICAL tests to decarburize blast furnace metal with pure oxygen were started at Domnarvet, Sweden, under the direction of Professor Bo Kalling as early as 1948. Using available high-phosphorus iron, it proved necessary to maintain a strong stirring and mixing effect to get the desired dephosphorization after decarburization. To bring about this intimate mixing between slag and metal, a rotating 3-ton furnace with a maximum speed of 40 rpm was used. By varying the rotating speed of this type furnace, it was found easy to carry out the reactions so that dephosphorization could take place, even for high-carbon steel.

About two years ago it was decided that the Domnarvet steel plant should have its annual ingot production increased by 100,000 tons, based on local ore as the raw material. This was the opportunity to prove the pilot-scale results in a full-scale production unit, and a 30-ton rotating furnace was built according to Stora Kopparberg's ideas by the Damag Co. in Germany. Since the start in May, 1956, the design of the furnace has proved satisfactory.

The furnace body, which rotates at a maximum speed of 30 rpm, rests on four wheels within a frame that can be turned on trunnions for different positions of charging, blowing, and tapping. The vessel is about 18 ft long with an outside diam of 11½ ft. In blowing position, the furnace is inclined 15° to increase furnace volume and protect the bottom from overheating. The oxygen is blown into the furnace above the bath surface.

The Kal-Do Heat Cycle

This process (named after the originator, Kalling, and the place, Domnarvet) is being used at Domnarvet to decarburize high-phosphorus iron, about 1.8 pct P, to steel with low S, P, and N. The main part of the phosphorus is oxidized before the iron reaches 1.5 pct C.

The large excess of heat, which is obtained in the process, is utilized for reduction of iron directly from ore, although it could also be used for melting scrap in areas where scrap is plentiful. Most of the ore and lime additions are made before charging the hot metal, and further additions are made after the phosphorous slag has been tapped.

It has been found to be advantageous to tap the phosphorous slag twice. As a rule, the first slag-off will be at 0.2 to 0.3 pct P, which analysis can be obtained with only 3 pct total iron in the slag. At the second, smaller slag-off, which is done at about 1 pct C, the phosphorus is usually less than 0.1 pct. Together, these two phosphorous slags amount to 300 to 400 lb per net ton of blast furnace iron. They contain, on the average, 16 to 18 pct P_2O_5 and 5 pct Fe and are sold for fertilizing purposes. Except for these two stops for tapping phosphorous slag, there is usually one additional stop for temperature and carbon control when the latter is 0.10 to 0.20 pct above the objective. The tapping temperature can be kept within very narrow limits.

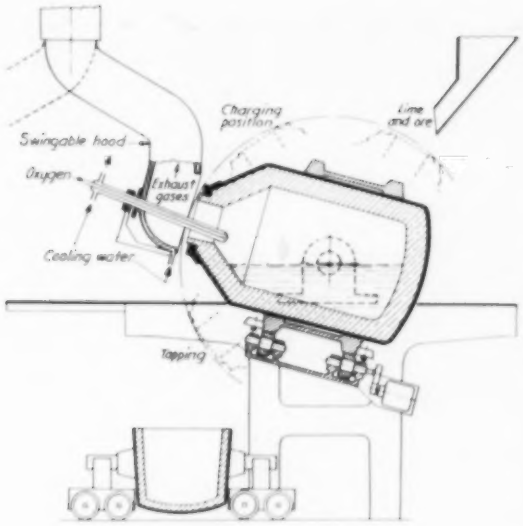
Kal-Do on low-phosphorus hot metal

The Kal-Do process is independent of hot-metal analysis. It has, in fact, certain distinguishing characteristics which are important for those plants utilizing low or medium phosphorus hot metal.

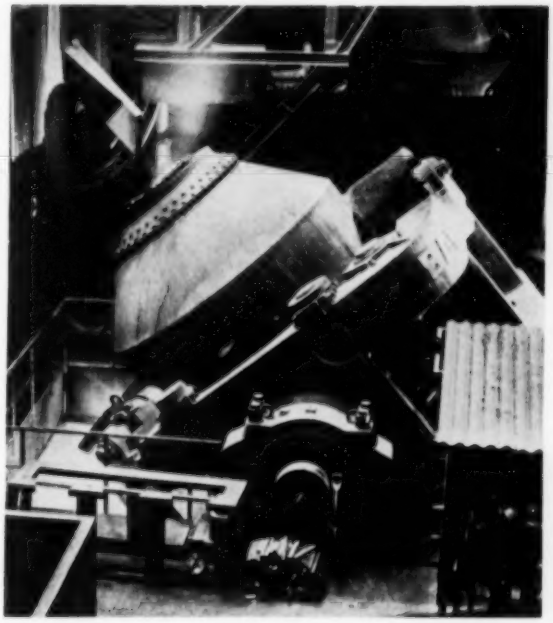
Using American-type hot metal of 0.1 to 0.3 pct P and 1 pct Si, it has been found feasible to add only as much lime with the hot metal to give a slag basicity less than 1.5 ($CaO + MgO/SiO_2$) after most of the silicon has been oxidized. At this basicity a fluid slag can be obtained at 1500°C without FeO or other fluxing materials. With the possibility of approaching equilibrium between steel and slag due to the high rotating speed in the Kal-Do process, the FeO content in the slag can be reduced to only 2 or 3 pct before slag-off. Thereafter further lime and ore are added, and carbon is brought down to the desired content. On these heats a stop is also made to control temperature and carbon before the final blowing.

The first slag is not basic enough to bind phosphorus and sulfur, but the required dephosphorization and desulfurization can be obtained with lime

FOLKE JOHANSSON is Assistant to the Director of Research at Stora Kopparbergs Bergslag A/B at Domnarvet, Sweden. This paper was presented at the AIME Annual Meeting, New Orleans, February 1957.



Above is shown a diagram of the furnace body, with the positions for charging, blowing, and tapping indicated. The vessel is about 17 ft long and has an outside diameter of 11½ ft.



Here the furnace is shown in charging position.

added after the silica-rich slag has been tapped off. To bind the phosphorus this slag does not even have to be melted. The main part of the basic, end slag, which contains about 15 pct Fe, can be returned to the process and reused.

On experiments with a medium phosphorus iron containing 0.27 pct P, both a silicate slag and a phosphorus-rich slag were tapped off to insure a low phosphorus content. Unfortunately there has not been sufficient opportunity to make more than occasional heats with this kind of hot metal, because for these tests cold pig iron must be remelted in an electric arc furnace. With more experience the results will, of course, be improved.

Raw material considerations

Application of the Kal-Do process to pig iron with high phosphorus content has already been discussed. In Europe, the basic bessemer hot metal can be produced $4 to $6 per ton cheaper than ordinary open-hearth hot metal. Furthermore, the basic bessemer slag will credit the process with some $4 per ton of steel. But with hot metal of more than 0.2 pct P, it is easier to bring the phosphorus down in the Kal-Do rotating furnace than in stationary converters.

Ore or sinter can advantageously be used for cooling instead of scrap. According to Stora Kopparberg's experience, such a high quality ore for decarburization as is required in open hearths or electric furnaces, is not necessary for the Kal-Do process. At Domnarvet, fines screened from ordinary blast furnace sinter are used. Continuous charging of small sized ore, concentrates, or sponge iron can be done during the blowing stages and should produce real advantages. So far this has been tested only in small scale operations.

Only burned lime is being used for slag formation, but it would naturally be possible also to use limestone. No fluxing materials are being used in the process at Domnarvet.

Heat economy of the process

The greater proportion of the carbon in the hot metal is burned to CO_2 in the Kal-Do process; there is thus considerably more heat developed than in other oxygen processes.

Using hot metal of 3.6 pct C, 0.3 pct Si, 0.7 pct Mn, and 1.9 pct P, an average of 300 lb of 55 pct Fe sinter is melted, the hot metal temperature being only about 1250°C. This is in addition to 280 lb of lime with up to 20 pct ore. For hot metal with about 4 pct C, 1 pct Si, and low phosphorus, some 240 to 280 lb of sinter has been used for cooling. When shorter heat cycles and higher hot metal temperatures prevail, still more iron can, of course, be reduced directly from ore.

Experience has not yet proven how much scrap can be melted instead of ore, but at least 40 pct of the weight of hot metal is undoubtedly possible and practical.

Steel yield

Kal-Do offers the possibility of high ingot yield, but unfortunately there is too much scrap in the slag pots and on the pit side to obtain thoroughly satisfactory results. However, on a number of control heats the following figures were obtained:

Input		Output	
Hot metal	2000 lb	Approved ingot	1942 lb
Fe from ore	160 lb	Scrap	54 lb
Alloys	20 lb	Alloy losses	130 lb
		Fe in slag and fumes	54 lb
Total	2180 lb	Total	2180 lb

Metallic yield, 91.6 pct; approved ingot yield, 89.1 pct.

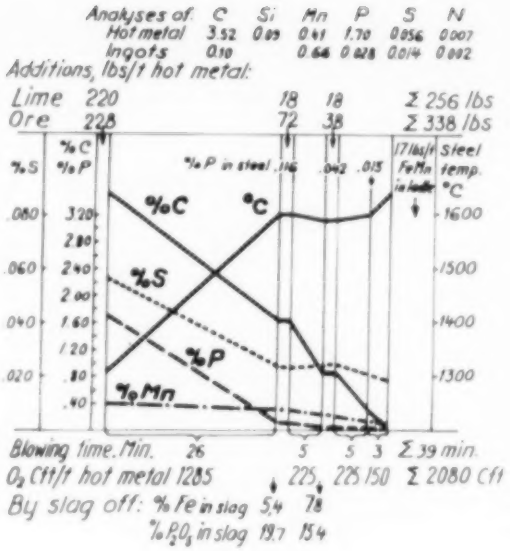
To further improve the yield, the end slag will be returned. In this manner the lime and phosphorus content can be transferred to valuable thomas phosphates with 20 pct P_2O_5. The steel yield would then be from 1 to 1.5 pct higher.

Using normal basic open-hearth hot metal with 3.7 pct C, 1.2 pct Si, 1.0 pct Mn, and 0.1 pct P, the alloy losses are 0.5 pct less, and the slag losses are less too, because of reduced slag volume. As high as 93 pct metallic yield could, therefore, be expected. The reason for this good yield is mainly the very low iron content in the slag, which has been tapped off after the silicon oxidation, and the relatively low fume losses.

Fume aspects

In the Kal-Do process fume losses are much less than in other oxygen processes. The reason is that the oxidation, as in the open hearth, takes place mainly via the slag. Most of the fume loss will occur before the slag is formed, but even then it is insignificant compared to other processes. Fume may be effectively reduced by adding a small amount of water, about 2 gallons per ton, to the oxygen. This amount of water corresponds to less than 1 pct ore, comparing the cooling effect.

In those locations where it is necessary to clean the exhaust gases, one can calculate lower costs, with the Kal-Do process than with other processes. This does not only refer to smaller amounts of dust and larger particle size, but also to the lower air requirements due to the low carbon monoxide content. The average heat content, including combustion heat, is measured at 400 Btu per min, which is only about one fifth of that in a stationary oxygen converter of comparable size. The gas volume, which has to be cleaned, varies with this heat content.


Analyses of: C Si Mn P S N
Hot metal 3.52 0.09 0.41 1.70 0.056 0.007
Ingots 0.10 0.64 0.028 0.014 0.002
Additions, lbs/t hot metal:
Lime 220 18 18 Σ 256 lbs
Ore 228 72 38 Σ 338 lbs
%C
%S %P
%P in steel .116 .042 .013
17 lbs/t FeMn in ladle
Steel temp. °C
%C
°C
%S
%P
%Mn
.080 3.20 .060 2.40 .040 1.60 .020 .80
2.80 2.00 1.20 .40
1600 1500 1400 1300
Blowing time. Min. 26 5 5 3 Σ 39 min.
O2 Cft/t hot metal 1285 225 285 150 Σ 2080 Cft
By slag off: % Fe in slag 5.4 7.8
% P2O5 in slag 19.7 15.4

The Kal-Do process is quite independent of the hot metal analysis; here are the results of tests on a low phosphorus iron similar to that encountered in the U. S. During the early stages of the blow some phosphorus was picked up from the furnace lining.

Furnace lining

The working lining in the Domnarvet furnace is 14 in. tar dolomite bricks with about 2 in. rammed dolomite between these bricks and the back lining. With the high phosphorus iron at Domnarvet, relining has to be done at the present time after about 50 heats. With 14-in. magnesite bricks, 83 heats were obtained, but in Sweden these bricks are three times as expensive as dolomite bricks. It was decided, therefore, to continue using dolomite, but through an improved lining technique it is expected to be able to wear the bricks thinner and thus be able to obtain at least 60 melts. The comparatively low lining life is mainly caused by the large slag volume and the high temperature which is obtained at the carbon monoxide combustion. This seems to be explained from the fact that the maximum wear of the lining is influenced by the direction of the oxygen stream. Mechanical wear due to rotation is insignificant.

Insufficient experience is available to determine the wear of the lining from the use of ordinary open-hearth hot metal. However, by slagging off the silica rich slag at 1500 C, the lining life will be much longer than with high phosphorus iron, due to shorter blowing time and smaller slag volume.

With larger furnaces, a better wear resistance is to be expected. In any case, the wear is much less in the 30-ton furnace than it was in the 3-ton experimental furnace.

Steel quality

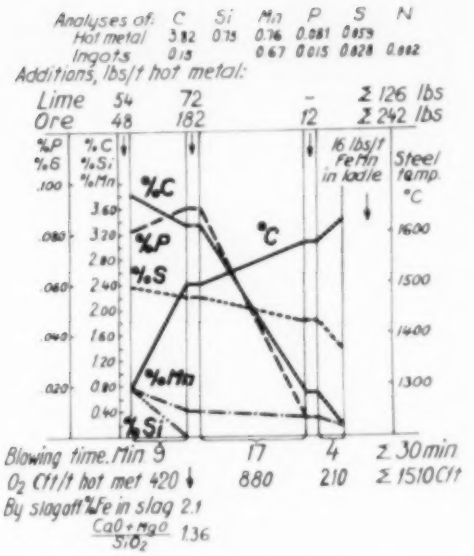
As already mentioned, there are no difficulties in producing steel with low phosphorus content, regardless of the phosphorus content of the hot metal. Specifications of a maximum of 0.025 pct P, using high-phosphorus iron, and a maximum of 0.015 pct P, using ordinary open-hearth quality iron can be met.

Using high phosphorus iron, good desulfurization can be achieved due to the large slag volume, for example from 0.078 pct to 0.019 pct S as a weekly average. This sulfur is half that of basic bessemer steel produced from hot metal of the same sulfur content. This is explained by the fact that the formation of slag in the basic bessemer does not occur before the carbon is down to below 0.05 pct at the slag-off, compared to 1.5 pct C in the Kal-Do process. The same very efficient desulfurization can not be expected using low phosphorus hot metal. However, 50 pct desulfurization has been obtained in these heats.

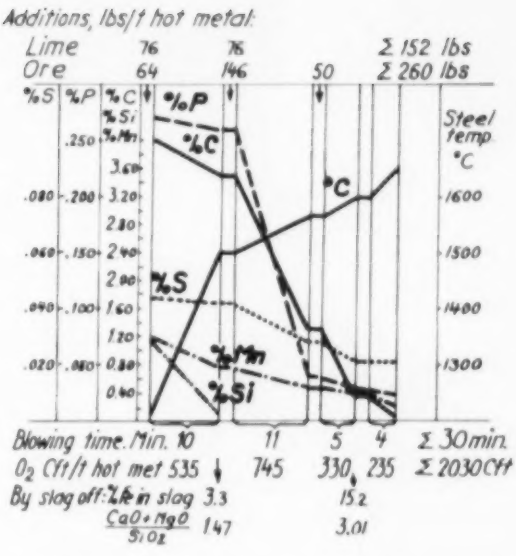
The nitrogen content is 0.002 to 0.003 pct on rimmed steel and 0.002 to 0.004 pct on killed steel. These values have also been obtained using only 95 pct pure oxygen. The process is, therefore, less dependent on high purity oxygen than other oxygen processes.

The oxygen content in the steel before deoxidation is the same as for ordinary open-hearth steel of the same carbon content. Through rotation without adding oxygen it is possible to reduce the oxygen in the steel even more, so that the content will be close to equilibrium. It is important for steel quality that the tapping temperature be kept within narrow limits, and the process can be completed and tapped at high temperatures without qualitative disadvantages.

At Domnarvet mostly rimmed steel has been produced thus far; it has proved to be especially suitable for deep drawing and wire drawing. Steel with a very low carbon content, for example a maximum of 0.02 pct C, can advantageously be produced in the Kal-Do furnace and can also be hot rolled at low manganese contents, due to low sulfur analyses.


Analyses of: C Si Mn P S N
Hot metal 3.82 0.75 0.76 0.081 0.053
Ingots 0.13 0.67 0.015 0.028 0.002
Additions, lbs/t hot metal:
Lime 54 72 - Σ 126 lbs
Ore 48 182 12 Σ 242 lbs
16 lbs/t FeMn in ladle
%P %S %C %Si %Mn
Steel temp. °C
Blowing time. Min 9 17 4 Σ 30 min
O2 Cft/t hot met 420 880 210 Σ 1510 Cft
By slag off: %Fe in slag 2.1
CaO+MgO / SiO2 1.36

A Kal-Do blow on a medium-phosphorus hot metal charge containing 0.27 pct P. A silicate slag and later a phosphorus-rich slag were tapped. A much lower phosphorus content might have been achieved through additional lime.


Additions, lbs/t hot metal:
Lime 76 76 Σ 152 lbs
Ore 64 146 50 Σ 260 lbs
%S %P %C %Si %Mn
Steel temp. °C
Blowing time. Min. 10 11 5 4 Σ 30 min.
O2 Cft/t hot met 535 745 330 235 Σ 2030 Cft
By slag off: %Fe in slag 3.3 15.2
CaO+MgO / SiO2 1.47 3.01

The Kal-Do process applied to a typical high-phosphorus iron encountered at the Domnarvet plant in Sweden. It results in a steel of low S, P, and N content. Thus far, mostly rimmed steels, especially suitable for deep drawing and wire drawing, have been produced.

A large number of semi-killed heats have been made for plates. The very low nitrogen and phosphorus contents advantageously influence weldability and impact tests at low temperatures. These steels, which previously were made in electric arc furnaces, are expected to dominate the Kal-Do furnace at Domnarvet.

This process is well fitted for production of killed steels of any carbon content. In making high carbon steels the carbon can, as in the open hearth, easily be caught at the desired level and recarburization in the ladle is not necessary. The procedure is usually to presample at 0.1 to 0.2 pct C above the desired carbon content. The right moment for this test is easily estimated from the oxygen consumption.

Production

The production capacity at Domnarvet is at present 29 tons of steel per heat and 15 heats per day, blowing high phosphorus iron; this corresponds to 18 tons per hr. The time from tap to tap is consequently 96 min, of which 40 to 45 min is the effective blowing time. The rest of the time can, of course, be reduced considerably in a new plant when using better arrangements for charging and testing. Blowing time for low phosphorus iron will, however, be below 30 min, judging from about 20 heats that have been made; the time for slag-offs and testing is also less.

The following figures for two size furnaces in newly erected plants, using low phosphorus iron, can be foreseen:

Furnace capacity, net ton		20	100
Charging time	min	10	15
Blowing	min	30	45
Slagging and testing	min	20	30
Total time	min	60	90
Production tons per hr		29	67
Production tons per year (7500 hr)		215,000	500,000

These figures presume one extra vessel for relining while the other is operating. The necessary crane capacity to change the 30-ton vessels is 135 tons which will add to the cost of the building and foundations.

Economic aspects

Regarding operating costs, the present somewhat high cost of lining has to be weighed against high yield and heat economy, flexibility in operations, and especially the possibility of utilizing cheaper raw materials. Remaining costs would be similar to those in comparable processes. A complete evaluation of operating costs will, of course, be necessary in every case.

The investment cost for the Kal-Do furnace will be higher than for stationary converters for the same output, due to the fact that larger units will be needed and the rotating device is extra. However, as the gas cleaning equipment can be made considerably cheaper than for a standard stationary converter, we do not believe that total investment cost will be higher for the whole plant.

Summary

The Kal-Do process has been developed to convert high-phosphorus iron into steel of open-hearth quality using high purity oxygen. However, due to some characteristic features, such as high yield and outstanding heat economy, the process will also be economical in the use of low-phosphorus iron. Of great importance is the fact that large amounts of ore can be used as a cooling material instead of scrap, and that the process can be used for all types of blast furnace or cupola iron in making all specifications of high or low grade carbon steels.

Arc Melting of Reactive Metals

Condensed from an article
by J. L. Ham and C. B. Sibley

OF the various methods devised for melting and refining reactive metals, arc melting in a water-cooled copper pipe has undoubtedly contributed much to recent advancements. In the *cold mold* method, a strip of metal is continuously formed in a tube containing the charge, then arc melted under a slag in a water-cooled mold.

The cold mold arc process is extremely versatile and can handle almost any metal or alloy, regardless of its melting point or degree of reactivity.

Cold mold furnace design

Fig. 1 shows a design of a permanent electrode cold mold arc furnace. Fig. 2 shows two consumable electrode cold mold furnaces with different drive mechanisms. Selection of drive depends on head room and type of electrode. Various electrode forming methods for consumable electrode furnaces are illustrated in Fig. 3.

Skull furnaces

Simultaneous with the evolution of cold mold arc furnaces, *skull* furnaces were introduced and tested by NRC, Rem Cru, and others. The term *skull* signifies that a liquid pool of the metal is contained in a solid skull of the same metal. This is maintained by control of relative rates of power input to the liquid and heat loss through the skull. The skull furnace has come to be associated with furnaces from which the charge is poured through the bottom or by tilting. Skull furnaces may be either the permanent or consumable electrode type. Since the charge to be poured is all molten at one time, alloy homogeneity does not depend on the electrode homogeneity or on the precision of any alloy metering device. Therefore, if stirring is provided, alloy homogeneity is assured, even in large furnaces. Another advantage of skull furnaces is that they can be used to make shaped castings.

J. L. HAM and C. B. SIBLEY are Research Associate and Furnace Section Manager, respectively, in National Research Corp. (NRC), and its subsidiary, NRC Equipment Corp. Paper presented at AIME Regional Reactive Metals Conference, Buffalo, March 1956.

Large water-cooled copper furnaces of either the cold mold or skull type are usually designed for remote control operation behind barriers. There is considerable interest in the use of permanent electrode skull furnaces for casting homogeneous electrodes for vacuum remelting prior to shape casting. To obtain sound castings from an argon atmosphere furnace it is often necessary to vacuum anneal the charge or pump down one or more times to remove hydrogen. To avoid glow discharge, power must be shut off at about 30 mm during pump down.

Among the most advanced types of skull furnaces are those operated by the U. S. Bureau of Mines in Albany, Ore. These are water-cooled copper consumable electrode furnaces, one bottom pour and one tilt pour.[1] These were never known to operate successfully in vacuum. A pressure of 30 mm proved most effective. As reported, the chloride, water, or hydrogen content of the melting stock was too high compared to pumping capacity to permit operation in vacuum below the glow discharge region. It is reported that castings of very impressive size, soundness and surface quality have been produced in these furnaces. (*see p. 971.*—Ed.)

Skull furnace design

Fig. 4 shows a drawing of a production-type skull furnace at the NRC laboratory.

Air lock valves on the feeder and mold drive mechanisms permit the furnace of Fig. 4 to be used for many heats without cooling the skull or opening the main tank. The electrode drive mechanism is designed for rapid withdrawl, to minimize cooling of the heat before pouring.

Inert atmosphere vs vacuum

In general, vacuum melting is to be preferred over argon melting, since it permits hydrogen removal and, in some cases, deoxidation by carbon.

Whether a given alloy component will require argon to prevent excessive vaporization depends on its vapor pressure, the melting point of the alloy or base metal, and the compound forming tendency.

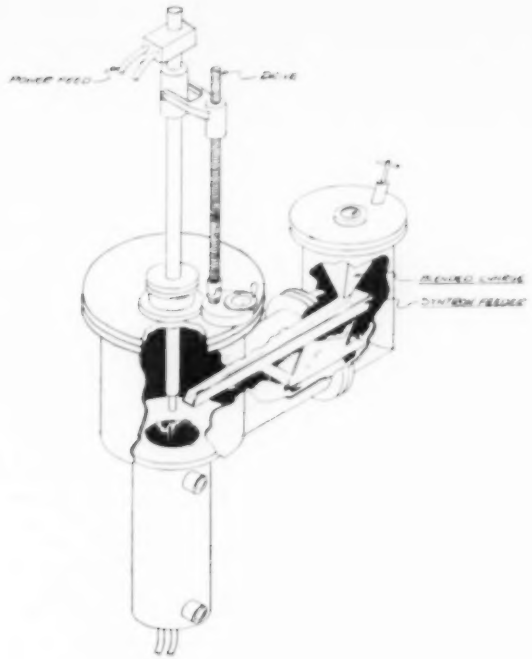


Fig. 1—Permanent electrode cold mold arc furnace with a syntron feeder. Other types use a long belt-type feeder.

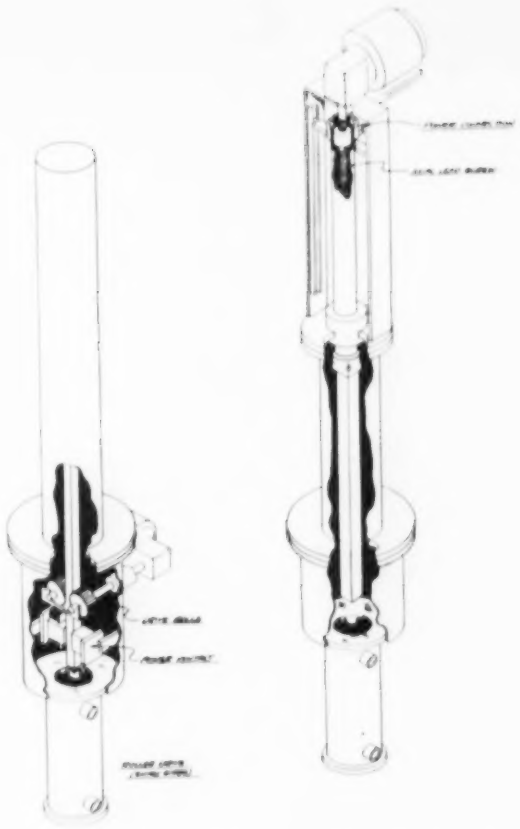


Fig. 2—Two types of consumable electrode cold mold furnace, with different drive mechanisms. Selection generally depends on type of electrode and available head room.

Titanium containing 4 to 8 pct of comparatively volatile metals such as aluminum or manganese can be vacuum melted. Only a few tenths of a percent of metals such as iron, cobalt, or nickel, and practically no aluminum, manganese, or chromium can be retained in molybdenum in vacuum arc melting. However, titanium, vanadium, zirconium, columbium, tantalum, and tungsten can be retained in molybdenum with little or no loss in vacuum melting, and without interfering with carbon deoxidation.

Even when argon must be employed in order to use permanent electrodes or to prevent vaporization of some volatile, it is highly desirable to evacuate the furnace prior to admission of argon. If the furnace walls become coated with manganese chloride or other hygroscopic material it is a good idea to heat the walls with steam or hot water as well.

Power partition and melting efficiency in permanent electrode melting

Although most types of permanent electrodes work best in the range of 200 to 760 mm, at least one furnace on the market is claimed to be operable in vacuum. Using an efficiently cooled large diameter cathodic molybdenum or tungsten electrode and a magnetic stirring coil to collimate the arc, it is possible to provide enough power at the bath to maintain a shallow pool in vacuum. However, the combination of a cold electrode and low pressure make for a very inefficient partition of power between electrode and bath. As pressure decreases near the glow discharge region a large proportion of power goes into the electrode. With tungsten or graphite electrodes this is apparent from the attendant increase in electrode brightness and rate of erosion.[9]

A large, efficiently cooled copper electrode absorbed 65 pct of the total power. This is to be expected, since the voltage drop at the electrode surface increases as the temperature of the surface decreases, and since little power is transmitted from electrode to bath by thermal radiation. Losses through tungsten electrodes ranged from 5 to 20 pct in the pressure range 200 to 380 mm of argon, depending on current, electrode size, arc length, etc.

Good power partition is very important in permanent electrode melting in order to get the greatest possible bath volume without excessive electrode erosion. Industrial permanent electrode furnaces usually use direct current with electrode negative and 200 to 760 mm of argon or an argon-helium mixture.

Research by Titanium Metals Corp. in 1952[9] established the proportionality of electrode power loss to current for a given pressure and revealed a definite increase in melting efficiency with decreasing arc length. Tungsten electrodes were found to permit better melting efficiency than graphite.

The data for a 10 lb permanent electrode skull furnace show a slight but consistent increase in melting efficiency as pressure is increased from 50 to 200 mm, but they also show a considerable variation in kw-hr per lb at a given pressure (250 mm). Therefore, although variations in voltage, current, and arc length from heat to heat have a considerable effect, better efficiency can be obtained at 250 mm than in the range 50 to 200 mm. Also, erosion is much less severe at the higher pressure.

Table I. Cold Mold Consumable Electrode Arc Melting*

Metal	Electrode Cross Section, Sq In.	Mold Diam, In.	Amps	Volts	Pressure, Mm	Kw-Hr per Lb
Ti	28	10	9000	30	30 A	0.5
Fe	3.1	5	850	45	760 A	0.32
Zr	3.9	9	4450 ac	41	0.008	0.28
Mo	3.9	9	6000 ac	65	0.025	0.43
Hf	5.0	4	2400	32	63 A-63 He	0.42
W	0.2	1.5	1650 ac	21	0.025	0.39
Ta	0.3	2	2000 ac	36	0.025	0.80
U	4.9	4	2300	27	190 A-190 He	0.15
Zr-Base	1.5	4	1200	35	0.1	0.22
Ni-Base	1.2	4	1400	30	0.003	0.30
Cu-Base	1.1	4	1400	30	0.005	0.23
Ti-Base	1.2	4	940	31	0.07	0.27

* De electrode negative except as noted.

Pressure, bath volume, and superheat in consumable electrode melting

For production of ingots from pure metals, partition of power between bath and electrode is of small importance, and either ac, dc, argon, or vacuum may be used. When dc is used, especially in argon melting, it is desirable to have the electrode negative, since the highly intense cathode spot may cause welding to, or burning-through the best designed cold mold. The use of a stirring coil not only improves homogeneity, but rotates the arc itself and thus reduces the probability of mold burn-through. When an alloy is involved, or the melt is to be poured as in skull melting, power partition is important, since it affects final bath volume, superheat and homogeneity.

The U. S. Bureau of Mines report* represents almost the sole source of quantitative information on bath volume. For a given power input, bath volume increases as pressure decreases—above the glow discharge range—for either straight or reverse polarity. When using ac, pressure had little or no effect, so that change in bath volume with pressure cannot be ascribed to thermal conduction or convection by the gas. The only other conceivable way in which pressure could affect either melting rate or bath volume for a given power input would be to change the partition of power between electrode and bath. The puzzling question is why the partition should be changed in the same direction for either polarity! It is tentatively suggested that the difference in temperature between electrode surface and bath surface is responsible for this behavior. Considerable rectification, which must result from such a temperature difference, was indicated by oscilloscope patterns obtained during the melting of molybdenum with ac by Climax Molybdenum Co.

Table II. Theoretical Melting Energies

Metal	Melting Pt, °C	Theoretical Kw-Hr per Lb	Ratio of Melting Pt, °K, to Atomic Wt
U	1130	0.030	5.9
Hf	1975	0.062	12.6
Ta	2996	0.085	18.1
W	3410	0.095	20.0
Zr	1845	0.114	23.2
Ni	1455	0.138	29.4
Co	1495	0.143	30.0
Mo	2610	0.144	30.1
Fe	1539	0.174	32.5
Ti	1670	0.194	40.7
Be	1315	0.907	176.3

The Bureau of Mines report presents data intended to show a general increase of bath volume with melting rate regardless of pressure or polarity. It is presently believed that since pressure affects melting rate, it must also affect bath volume and superheat. All of the power, except a portion of that radiated from the electrode and the liquid bath, eventually gets into the bath and must contribute to either bath volume or superheat. The amount contributed to each will depend to some extent on the partition of power between electrode and bath, which depends markedly on pressure. The cathode to anode melt ratio increases to a maximum at 30 mm as pressure decreases, then falls off due to glow discharge.

For the same power partition between electrode and bath, a somewhat better ratio of bath volume to kw-hr should be obtained on vacuum melting below 1 mm, since 30 mm or more of argon is sufficient to cause considerable loss by thermal conduction and convection in addition to radiation.

NRC data on kw-hr per lb for consumable electrode melting of titanium indicate a somewhat higher proportion of power to bath in vacuum melting than in argon melting. In consumable electrode skull furnaces, the power to the bath contributes more to superheat than to melting, as this heat must diffuse to the bottom of the bath to cause melting. Therefore, it seems probable that the vacuum method is superior for making castings, both from the standpoints of soundness—hydrogen content—and mold filling—superheat.

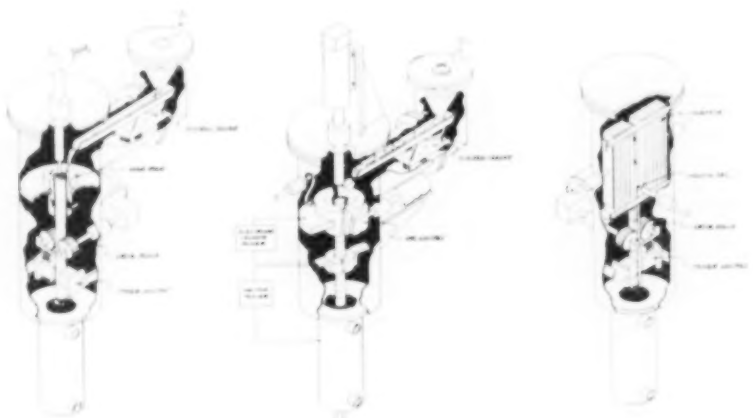


Fig. 3—Electrode forming methods for consumable electrode arc furnaces. Left, electrodes are formed in a cold mold; center, electrodes are welded from pellets or powder, and at right, the electrode is welded from short pieces held in a magazine.

Comparison of actual and theoretical energy per pound for the melting of various metals

In cold mold consumable electrode arc melting the energy required per pound of metal melted depends on the heat of fusion and the average specific heat from room temperature to perhaps 100°C above the melting point. The portion of the arc energy going directly into the bath increases the bath volume existing at any one time, but it cannot contribute to melting except as thermal radiation from the bath to electrode may contribute. Similarly, the rate of heat loss from the ingot is important so far as bath volume is concerned, but can have little to do with the melting rate at a given power level. The electrical resistivity of the electrode affects the kw-hr per lb insofar as it affects the loss by thermal radiation from the electrode.

Table I gives observed values for ten different metals for various mold sizes, atmospheres, and pressures, and Table II gives approximate theoretical values. The values of Table II were obtained by adding the heat of fusion to the heat content of the solid at the melting point minus that at 25°C.

There is little correlation between actual and theoretical kw-hr per lb, and it is apparent that such data are useful only for predicting the power requirements for melting different metals under a given set of conditions. Electrode resistance must also be taken into account.

Table II, also gives values for the ratio of melting point to atomic weight and this parameter is a very good index of the theoretical kw-hr per lb required for melting. This arises from the fact that most metals have the same entropy of melting and the same molar specific heat at elevated temperatures. Apparently the molar heat content of the liquid at the melting point (minus that at 298°K) is more accurately predicted by the combination of these rules than are the specific heat and heat of fusion by the individual rules. It can also be shown that the theoretical kw-hr per mol is proportional to melting point.

Defects in cold mold ingots

Consumable electrode melting can produce sound ingots with good surface from most metals and alloys. In large heats or where a high melting rate is used, the bath volume is large enough to cause serious piping if power is not tapered off at the end of the heat. Poor surface can result from too low power for a given mold size, or from permitting the arc to become too long. In vacuum melting, cold shuts formed under such conditions may be covered with an apparently sound crust when volatile metals are present in excessive amounts. However, when volatile metals are not present, vacuum melting produces smoother ingots than argon melting. Surface defects can be removed either by machining or by applying an arc to the surface while the ingot is rotated in an argon filled chamber.[25]

The cooling ingot shrinks and is in contact with the cold mold only directly opposite the arc. Cooling is by radiation in the case of vacuum melting and by radiation and gaseous convection in the case of argon melting. Therefore, the rate of cooling is not particularly drastic. Nevertheless, steep thermal gradients exist and there is a limit to the amount of hardening alloying elements which can be added without causing the ingot to crack.

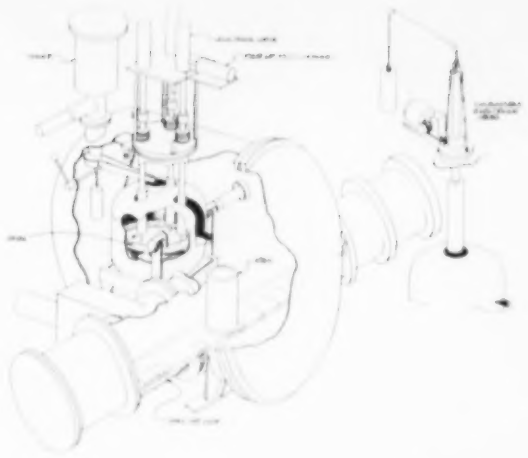


Fig. 4—Production type skull furnace design. Air lock valves permit many heats to be tapped without opening the main tank.

Minimum pumping speed for consumable electrode vacuum melting

The following remarks and sample calculations are idealized in that it is assumed that only one type of gas (carbon monoxide or hydrogen) is evolved by the metal and no gas is evolved by furnace parts, so the pump speeds calculated are minimum values. In the case of molybdenum, evolution of gases other than carbon monoxide from both the molybdenum and the furnace parts usually necessitate a pump speed about double the calculated value. Actual required speed for remelting of titanium should be very close to the calculated minimum, since only hydrogen is evolved from the metal. Iron, nickel, and cobalt-base alloys represent intermediate cases in which carbon monoxide, hydrogen, and water all occur in the exit gas.

For consumable electrode furnaces the calculation of minimum pumping speed required to maintain a given pressure or degree of purity is quite simple, once the required data as to melt stock analysis and melting rate are known.

For carbon deoxidation the required pumping speed is simply the initial oxygen content expressed as atm cu ft CO per lb multiplied by the melting rate in lb per min and divided by the final pressure in atmospheres. The partial pressure of carbon monoxide which can be tolerated depends on the amounts of carbon and oxygen that can be tolerated in the final product and must be evaluated by experience. This method would apply to molybdenum, tungsten, columbium, tantalum, vanadium, iron, nickel, cobalt, and their alloys. In the case of molybdenum, at least 0.05 pct C can be tolerated but more than 0.0005 pct O_2 is liable to cause hot shortness. Total pressures below 50 μ are sufficient to obtain the indicated $[C][O_2]$ concentration product.

For removal of hydrogen, the speed calculation is somewhat different. In most metals, liquid or solid, the solubility of hydrogen varies as the square root of the hydrogen pressure. The minimum required speed would be that required if the hydrogen concentration in liquid and pressure of hydrogen in the furnace were in equilibrium, and is given by the following equation:

$$Q_{min} = S\,R\,\frac{(C_i - C_f)}{(C_i)} \qquad [1]$$

where Q_{min} is the pumping speed in cfm at furnace port, S is the solubility of H_2 in the liquid at 1 atm H_2 pressure-atm cu ft per lb, R is the melting rate in lb per min, and C_i and C_f are the initial and final hydrogen concentration, respectively, in atm cu ft per lb.

Eq. 1 was derived by dividing T by P where

T = pump throughput-atm cfm

$$= \frac{R}{N}(C_i - C_f)$$

P = total pressure-atmospheres

$$= \frac{C_f}{S\,N} \qquad [2]$$

N = mol fraction of hydrogen in exit gas.

It is interesting to note that the mol fraction cancels, and that even when C_i is large compared to C_f the pumping speed must be quadrupled to reduce C_f by half. This equation is applicable to operations such as the vacuum degassing of steel as well as to consumable electrode melting. For pure iron the value of S is about 0.005 atm cu ft per lb.

So far as can be determined, no values have been published for titanium. The following steady state data on a titanium scrap heat were used to get a tentative value of S for hydrogen in titanium.

R = 1.7 lb per min
C_i = 0.016 pct = 0.0286 atm cu ft per lb
C_f = 0.003 pct = 0.0054 atm cu ft per lb
Q = 60 cfm (1 mm at furnace port).

The observed maximum steady state operating pressure for this heat was 1.04 mm. Substitution of this value in Eq. 2 shows that the pressure is consistent with the solubility value given below only if N = 0.5. This seems low and indicates considerable out-gassing of furnace parts or a higher than normal leak rate. However, this does not affect the accuracy of the following calculation of S.

Solving Eq. 1 for S using these values gives:

S = 0.210 cu ft per lb (0.117 pct).

Note that this value of S is not dependent on the accuracy of any pressure measurement during the run, but only on the accuracy of pumping speed measurement on the cold furnace.

The accuracy with which this value of S represents the solubility at 760 mm pressure depends on the validity of the $p^{1/2}$ solubility relationship from 1 to 760 mm. However, the relationship is probably valid in the pressure range of interest (1 μ to 1 mm) so that apparent S values, so derived, represent the constants required for pumping speed calculation. Variation of bath temperature with melting conditions probably has only a small effect on S.

Summary

For the production of ingots of reactive metals, the cold mold arc melting method has proven to be both practical and versatile. The trend is toward the use of vacuum rather than argon and of consumable rather than permanent electrodes. Permanent electrode skull furnaces for casting of electrodes may replace sponge blending and pressing for certain types of melting. A pressure of 250 mm of argon is recommended for permanent electrode operation.

Cold mold consumable electrode melting has been applied to at least ten different metals and their alloys. Energy per mol required to melt such metals is theoretically proportional to their melting points, but in practice, it varies widely with electrode resistance, pressure, and mold size.

Melting in vacuum, below the glow discharge pressure range, appears to be technically superior to argon melting, but is practical only with consumable electrodes low in volatile matter. Argon is required in some cases to retain volatile alloying constituents. Furnace evacuation is recommended even though inert gas at one atmosphere pressure is to be used for melting. Cold mold arc melting provides automatic hot topping and generally good ingot surface and quality. Vacuum melting provides better surface than argon melting.

Pumping speed requirements for consumable electrode vacuum arc melting can be estimated by calculating the minimum pump speed from the melting rate, hydrogen solubility, and initial and final hydrogen concentration. The solubility of hydrogen in liquid titanium is of the order of 1200 ppm at 760 mm pressure of hydrogen.

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Thermodynamics of the Cu-Fe-S System At Matte Smelting Temperatures

by W. A. Krivsky and R. Schuhmann, Jr.

PREVIOUS papers in this series on the thermodynamics of copper-smelting systems have presented a survey of the field and an outline of the overall program,[1] thermodynamic studies of iron silicate slags,[2-4] and thermodynamic data for simple mattes in the Cu-S system.[5] Since this program was started, an excellent summary of the whole field of physical chemistry of copper smelting was published by Ruddle.[6] The present paper represents a further step in the study of liquid mattes, and deals specifically with the Cu-Fe-S system. The importance of the Cu-Fe-S system to copper smelting is evident from the fact that these three elements constitute 90 to 99 pct of the total weight of most commercial mattes. In copper mattes oxygen is an important fourth component. Oxygen was rigorously excluded in the work to be described in this paper. However, the behavior of oxygen in mattes was the subject of a companion research by Lander[7] which will be described in a subsequent publication.

Several studies have been made previously of phase equilibria in the Cu-Fe-S system,[8-10] so that the ternary phase diagram is fairly well established in most regions. Also, separate studies have been made of the phase diagrams and thermodynamic properties for the three binary systems: Cu-S,[5,10-13] Cu-Fe,[10,14-17] and FeS.[11-14,18-22] However, no work has

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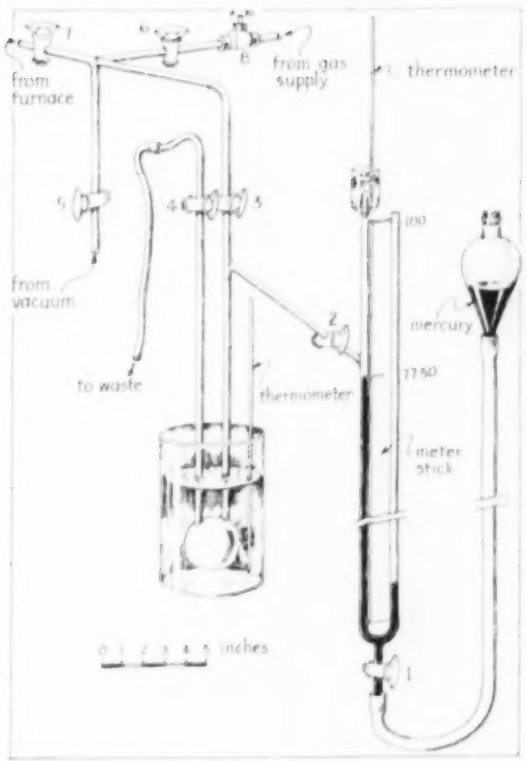

Fig. 1—Gas analysis apparatus.

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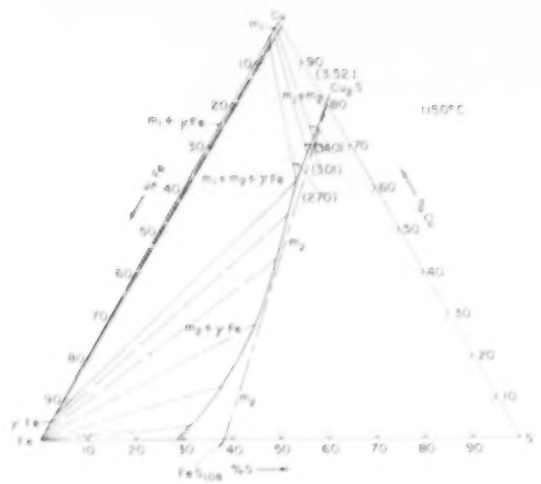

Fig. 2—Isothermal section of Cu-Fe-S system at 1150°C.

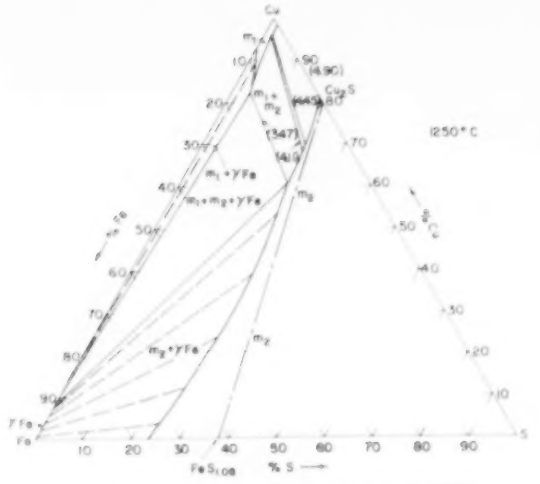

Fig. 3—Isothermal section of Cu-Fe-S system at 1250°C.

been reported to date on the thermodynamic properties of the ternary liquid mattes in the Cu-Fe-S system.

The experimental part of this study consisted of measuring H_2S:H_2 gas ratios in equilibrium with various Cu-Fe-S mixtures in the temperature range 1150° to 1350°C. The equilibrium measurements were made in the following systems: 1) gas:liquid matte, 2) gas:liquid matte:liquid metal, and 3) gas:liquid matte:liquid metal:solid metal. Complete chemical analyses were made of the equilibrium phases so that the results furnish a redetermination of a substantial portion of the Cu-Fe-S phase diagram at 1150° to 1350°C. From the experimental measurements of sulfur activity, various other thermodynamic properties of the mattes were calculated. In particular, several forms of the Gibbs-Duhem equation recently derived[9] for ternary systems were used to obtain activities of Cu_2S, FeS, copper, and iron as functions of melt composition.

Experimental Methods

Materials—The copper and copper sulfide were the same materials as used previously by Schuhmann and Moles.[5] High purity iron was obtained from the National Radiator Co. (99.91 pct Fe, 0.015 pct C, 0.01 pct Cu) and from the Westinghouse Electric Co. (99.95 pct Fe, 0.04 pct O, 0.005 pct C). In addition, an Armco iron sleeve was used during the investigation of the equilibria between liquid mattes, liquid metal, and solid metal. Iron sulfide was prepared by reacting Mallinckrodt USP precipitated sulfur with iron, followed by melting in a hydrogen atmosphere. No special precautions were taken to avoid oxygen contamination of the stock materials, since the oxygen was automatically removed during the long equilibration runs with a stream of dry H_2 or H_2S-H_2 mixture bubbling through the matte.

Procedure—The experimental procedure and apparatus were substantially the same as in the previous work on the Cu-S system.[5] Closely controlled mixtures of hydrogen and hydrogen sulfide were bubbled through the liquid mattes in a closed furnace system. Entrant and exit gas analyses were made, and the entrant gas composition was altered until both the entrant and exit gas compositions were the same and remained so for several hours. Equilibrium conditions were checked by approaching the equilibrium gas composition from both sides. After gas-liquid equilibrium was attained, samples of the liquid matte were drawn up into 7-mm Vycor tubing, pulled to a capillary opening at the lower end. When two liquid phases were present, both phases were sampled separately after allowing the system to stand quietly for some time at equilibrium. The capillary tip of the Vycor sampling tube used for the lower liquid-metal phase was sealed before use and then the seal was broken on the bottom of the crucible.

The apparatus of Schuhmann and Moles for analyzing H_2S:H_2 mixtures by fractional distillation at liquid-nitrogen temperatures was redesigned to get better accuracy in analyzing low H_2S mixtures. The new design, shown in Fig. 1, permitted rapid and accurate analysis of gas mixtures ranging from 0.1 to 100 pct H_2S. In the range from 0.1 to 5 pct H_2S, gas analyses were readily reproduced within ±0.01 pct H_2S. The analytical procedure involved 1) collecting the gas sample into a fixed volume and reading pressure and temperature, 2) immersing the bulb in liquid nitrogen and pumping off the hydrogen, 3) evaporating the H_2S into the original fixed volume, and 4) reading pressure and temperature for the H_2S alone. For samples with less than 5 pct H_2S, the pressure of H_2S was measured after a standard compression using the McLeod-gage type of arrangement which can be seen at the top of the mercury manometer in Fig. 1. Not shown in the figure is a liquid nitrogen trap which was placed in the vacuum line between the vacuum pump and stopcock 5. The addition of this trap greatly improved the accuracy by preventing transfer of condensable substances from the pumping system back into the sample bulb during the hydrogen distillation at liquid-nitrogen temperature.

The experiments on equilibration of gas mixtures with three condensed phases, γ-iron alloy, liquid metal, and liquid matte, proved very troublesome. For these determinations an Armco iron sleeve was placed in the alumina crucible to insure saturation with γ-iron. However, difficulty was encountered in adjusting the composition of the charge to obtain all three condensed phases present at equilibrium. This difficulty stemmed from errors in the previously

Table I. Equilibrium Data

Point No.	Temperature, °C	Gas Analysis, Pct H_2S	p_{H_2S}/p_{H_2} x 10^{3*}	log p_{H_2S}/p_{H_2}*	Chemical Analysis, Pct Cu	Pct Fe	Pct S	Equivalent Ratios, E_{Fe}	E_S
				Gas—Liquid Matte Equilibria					
9	1148	0.325	3.27	−2.486	71.33	7.17	21.50	0.186	0.972
10	1155	4.55	47.7	−1.321	68.84	8.42	22.74	0.218	1.024
44	1153	100.00	2090	+0.301	69.27	7.04	23.69	0.188	1.101
21	1150	100.00	2090	+0.301	67.25	8.63	24.12	0.226	1.100
48	1158	1.175	12.0	−1.921	31.93	39.39	28.68	0.737	0.935
37	1148	1.63	16.6	−1.780	32.17	38.76	29.07	0.733	0.957
36	1148	2.62	26.7	−1.573	32.15	38.54	29.31	0.732	0.969
35	1148	6.66	71.6	−1.145	31.68	38.24	30.08	0.733	1.004
34	1151	13.65	155.5	−0.808	31.61	37.98	30.41	0.732	1.021
33	1148	25.06	325.5	−0.487	31.38	37.80	30.82	0.733	1.040
8	1263	0.453	4.54	−2.343	71.28	7.15	21.57	0.186	0.976
11	1247	4.46	46.2	−1.335	69.35	8.21	22.44	0.212	1.010
43	1253	100.00	1336	+0.126	69.27	7.08	23.65	0.189	1.098
20	1246	100.00	1336	+0.126	67.54	8.61	23.85	0.225	1.085
28	1258	1.55	15.75	−1.802	32.29	39.25	28.46	0.734	0.927
29	1250	3.00	30.8	−1.512	32.09	38.73	29.18	0.733	0.962
31	1252	6.77	71.6	−1.145	32.13	38.15	29.72	0.730	0.960
30	1249	15.13	170	−0.770	31.65	37.97	30.38	0.732	1.020
23	1266	26.10	320	−0.495	31.81	37.72	30.47	0.730	1.026
32	1247	25.76	312	−0.506	31.32	37.60	30.78	0.734	1.037
7	1349	0.556	5.60	−2.252	71.26	7.20	21.54	0.187	0.974
16	1357	0.712	7.30	−2.137	69.74	8.77	21.49	0.223	0.950
15	1354	1.30	13.6	−1.866	69.70	8.54	21.76	0.218	0.967
12	1349	4.06	41.2	−1.385	69.26	8.81	21.93	0.224	0.974
13	1353	7.30	75.1	−1.124	69.22	8.26	22.52	0.214	1.014
14	1353	7.46	76.9	−1.114	68.76	8.65	22.59	0.223	1.013
17	1364	24.45	282	−0.582	68.48	8.56	22.96	0.221	1.034
18	1349	49.99	516	−0.287	68.30	8.33	23.37	0.217	1.062
19	1348	100.00	923	−0.035	67.97	8.37	23.66	0.219	1.077
42	1354	100.00	923	−0.035	69.58	6.97	23.45	0.186	1.087
40	1350	0.564	6.00	−2.222	52.71	25.40	21.89	0.523	0.785
27	1346	2.24	22.7	−1.644	32.31	39.20	28.49	0.734	0.929
47	1361	3.94	40.0	−1.398	31.91	39.03	29.06	0.736	0.954
26	1351	5.69	57.95	−1.237	32.28	38.34	29.38	0.730	0.974
25	1353	11.11	116.1	−0.935	32.07	38.21	29.72	0.731	0.990
24	1347	25.75	256.5	−0.591	31.67	37.85	30.48	0.731	1.025
				Gas—Liquid Matte—Liquid Metal Equilibria†					
6	1154	0.0339	3.40	−2.468	71.38	7.30	21.32	0.189	0.960
					97.13	1.51	1.36	0.034	0.054
1	1150	0.299	3.01	−2.521	67.76	10.22	22.02	0.256	0.959
					96.43	2.34	1.23	0.052	0.048
5	1256	0.443	4.45	−2.352	69.65	8.59	21.76	0.219	0.967
					96.66	2.12	1.22	0.048	0.048
2	1251	0.409	4.11	−2.386	68.09	9.95	21.96	0.250	0.959
					95.76	2.63	1.61	0.059	0.063
4	1351	0.571	5.74	−2.241	70.21	8.29	21.50	0.212	0.957
					96.34	2.32	1.34	0.052	0.052
3	1351	0.550	5.53	−2.257	67.87	10.30	21.83	0.257	0.948
					95.55	3.03	1.42	0.067	0.055
45	1351	0.421	4.23	−2.373	54.91	23.07	22.02	0.489	0.812
					77.14	18.46	4.40	0.353	0.146
46	1357	0.418	4.20	−2.376	54.03	24.44	21.53	0.507	0.770
					65.49	28.04	6.47	0.493	0.198
				Gas—Liquid Matte—Liquid Metal—Solid Metal Equilibria†					
22	1150	0.265	2.66	−2.575	61.76	15.30	22.88	0.361	0.938
					93.10	5.06	1.84	0.110	0.070
38	1150	0.270	2.71	−2.567	61.49	15.62	22.89	0.366	0.935
					94.27	4.58	1.15	0.100	0.044
39	1285	0.378	3.79	−2.422	58.97	18.86	22.17	0.421	0.862
					75.05	20.40	4.55	0.382	0.149
41	1353	0.442	4.44	−2.352	50.26	29.69	20.05	0.573	0.674
					42.12	51.37	6.51	0.735	0.162

* Corrected for dissociation at furnace temperature.

† Corrected to 100 pct Cu + Fe + S.

‡ $E_{Fe} = \frac{n_{Fe}}{n_{Fe} + \frac{n_{Cu}}{2}}$, $E_S = \frac{n_S}{n_{Fe} + \frac{n_{Cu}}{2}}$.

§ For each point, the first line gives the analysis of the matte phase and the second line the analysis of the liquid metal phase.

available ternary diagrams used as a guide, from the narrowness of the three-phase region at higher temperatures, and from the great temperature sensitivity of the various mutual solubilities. Then, when the desired equilibrium conditions were approached, sampling of the liquids became difficult and even impossible, owing to the buildup of solid iron-alloy bridges and shells over the liquid phases in the crucible.

Experimental Results

Five experimental campaigns were carried out, each campaign representing from four to six weeks of continuous operation at temperature. Once the refractories were dried thoroughly and the charge became deoxidized at the start of the campaign, successive equilibrium determinations could be completed at from 24 to 48-hr intervals. Each equilibration involved adjustment of temperature and charge composition followed by repeated adjustments of entrant gas composition and repeated gas analyses to achieve the same composition in entrant and exit gases. Over 600 gas analyses were made during the five campaigns. All equilibrium points were approached from both directions; that is, by trying entrant gases both richer and leaner in H_2S than the final equilibrium gas mixture. After all the adjustments were completed, the system was usually operated for several hours (overnight) and rechecked to insure that no further change or reaction could occur.

Table I gives temperatures, gas compositions, and melt compositions for the 48 equilibrium determinations which were completed satisfactorily during the five experimental campaigns. For convenience

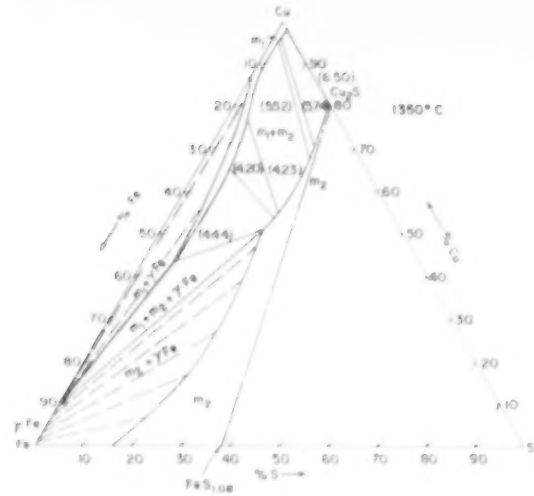

Fig. 4—Isothermal section of Cu-Fe-S system at 1350°C.

in subsequent thermodynamic calculations, the gas compositions determined analytically (column 3) were corrected for dissociation at furnace temperature, using Kelley's free energy data for H_2S.[17] The corrected H_2S:H_2 ratios and values of $\log_{10}(p_{H_2O}/p_{H_2})$ are given in columns 4 and 5. Separate analyses were made in duplicate for copper, iron, and sulfur, and the analyses appeared to be accurate and reproducible to within <0.05 wt pct of each constituent. The matte and metal analyses totalled 99.7 pct on the average, and so were corrected slightly to make copper plus iron plus sulfur equal 100 pct. R_2O_3 analyses (after iron removal) were never higher than 0.07 pct, and SiO_2 contents averaged 0.02 pct. The last two columns give melt compositions in terms of *equivalent ratios*, E_{Fe} and E_S. These ratios are based on the ordinary valences of the three components in the stoichiometric sulfides Cu_2S and FeS. E_{Fe} is the ratio of equivalents of iron to total cation equivalents while E_S is the ratio of equivalents of sulfur to total cation equivalents. For stoichiometric Cu_2S, $E_S = 1$ and $E_{Fe} = 0$; for stoichiometric FeS, $E_S = 1$ and $E_{Fe} = 1$; and for pseudobinary mixtures of Cu_2S and FeS, $E_S = 1$ and E_{Fe} = mol fraction FeS. These parameters proved more convenient than mol fractions of the elements in simplifying and correlating the data on liquid mattes.

The order in which the points were determined is indicated by the chronological point numbers in column 1 of Table I. To facilitate study of the results, the table is arranged in three parts, according to the number of phases present at equilibrium. In each part, the points are further grouped according to temperature, and at each temperature are arranged in order of increasing E_{Fe} and E_S.

Ternary Isotherms at 1150°, 1250°, and 1350°C—The analytical data in the last two parts of Table I determine a substantial portion of the Cu-Fe-S phase diagram for the temperature range 1150° to 1350°C. Figs. 2, 3, and 4 give isothermal sections of this diagram at 1150°, 1250°, and 1350°C, respectively. The liquid matte-liquid metal miscibility gap and the three-phase regions at each temperature were located reasonably well in the present work. The remainder of the diagram is based on the work of Schlegel and Schüller,[10] and on other published data for the three binary systems.[5,12,14] These diagrams also show the variations of sulfur activity in the miscibility gap, from the binary Cu-S two-liquid system over to the three-phase region. The figures in parentheses for each tie line give $p_{H_2O}/p_{H_2} \times 10^5$.

Within the temperature range under consideration, the Cu-Fe-S system is characterized by large regions of immiscibility. The two stoichiometric sulfides in the pseudobinary Cu_2S-FeS system are completely miscible and form liquid mattes covering the entire range from Cu_2S to FeS. However, reduction of the sulfur content for high-copper mattes leads to the formation of a separate liquid-metal phase high in copper. Reduction of the sulfur content of low-copper mattes leads to the precipitation of the γ-iron phase (with some copper in solid solution). Along the Cu-Fe side, these isotherms also show the immiscibility of the two metals copper and iron. Of particular interest in the 1350° isotherm is the long narrow single-phase region for liquid metal (m_1). At this temperature, the solubility of iron in liquid copper is multiplied several-fold by the addition of a critical percentage of sulfur to the liquid. As a result of the temperature sensitivity of iron solubility in liquid copper, the two-liquid miscibility gap region enlarges very rapidly at the expense of the three-phase region as the temperature is increased above 1250°C.

Sulfur Activities in Liquid Mattes—Fig. 5 summarizes the sulfur activity data for low-iron mattes ($E_{Fe} = 0.2$; about 70 wt pct Cu) and for high-iron mattes ($E_{Fe} = 0.73$; about 32 pct Cu), and also shows for comparison previous data[5] for mattes containing no iron ($E_{Fe} = 0$; about 80 pct Cu). These plots show that sulfur activity is very sensitive to sulfur content, especially in high-copper mattes. For example, at 1150°C, a relative change of only 5 pct in the sulfur content (from $E_S = 1.00$ to $E_S = 1.05$) causes about a tenfold change in H_2S:H_2 ratio, corresponding to a hundredfold change in p_{S_2}. Mattes approaching FeS in composition exhibit progressively less steep variations of sulfur activity with sulfur content.

The apparent scatter of the experimental points in Fig. 5 is due primarily to experimental error in the sulfur analyses of the mattes and not to errors in the gas analyses. The analytical error in sulfur determinations is in fact emphasized by the expanded E_S scale in these plots. Thus, as E_S varies from 0.96 to 1.06, the matte analysis changes by only about 2 pct S, while the H_2S:H_2 ratio changes by as much as a hundredfold in some instances.

As a result of the steep slopes of the curves in Fig. 5, small errors in the sulfur analyses produce large errors in estimates of the temperature coefficients of sulfur activity. Accordingly, it does not appear worthwhile to attempt to calculate heats of solution. However, the data do indicate semiquantitatively that the heat of solution of sulfur in liquid mattes tends to decrease rather markedly (more heat evolved on solution of sulfur) with decreasing sulfur content.

The general similarity of the three sets of curves for three different Cu-Fe ratios in Fig. 5 indicates that substitution of iron for copper in mattes causes no major or abrupt change in the measured properties of the matte solutions, when the substitution is made on the basis of one atom of iron for two atoms of copper. In other words, sulfur activity is determined primarily by the equivalent ratio E_S and by temperature, and is only secondarily affected by the Cu:Fe ratio or by E_{Fe}. Evidently the change in

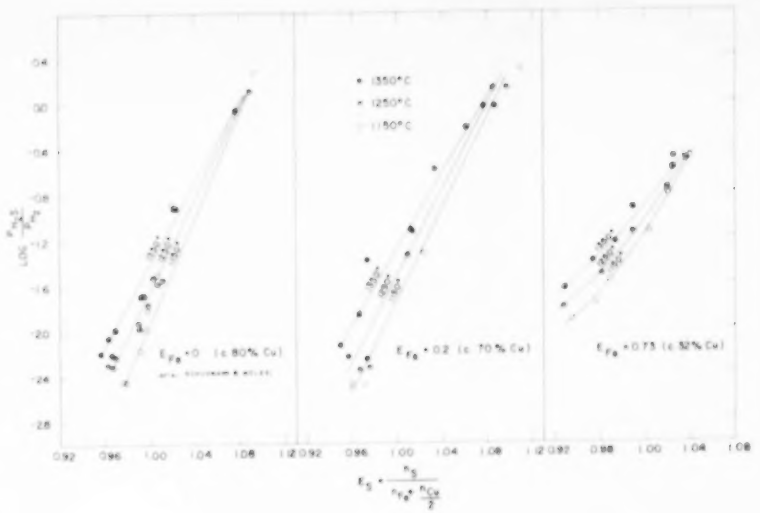

Fig. 5—Sulfur activities in liquid mattes.

metal-sulfur coordination which must occur with substitution of one iron for two coppers does not greatly affect the energetics of the solution structure.

The variation of sulfur activity with matte composition at 1350°C is shown in more detail in Fig. 6. Also included in this graph is the curve giving sulfur activities for mattes with compositions along the boundary of the miscibility gap. This curve is nearly horizontal, because sulfur activity varies little as the composition of the metal-saturated matte changes from the high-copper to the high-iron end of the miscibility gap. Moreover, the sulfur activity along the miscibility gap passes through a minimum when the metal-saturated matte has the composition corresponding to $E_{Fe} = 0.50$ and $E_S = 0.79$.

The dashed sulfur-activity curve for $E_{Fe} = 0.50$ in Fig. 6 was estimated by interpolation between the curves for $E_{Fe} = 0.73$ and $E_{Fe} = 0.2$, and by interpolating between the two points for $E_{Fe} = 0.489$ and $E_{Fe} = 0.507$ along the miscibility gap. The smoothed curves in Fig. 6 were taken as the basis for the Gibbs-Duhem integrations described in the next section.

Application of Gibbs-Duhem Equations

When the activity of one component of a ternary system is known over a sufficient portion of the ternary isotherm, the activities of the other components are readily calculated by integrating various modifications of the Gibbs-Duhem relation.[28] However, these integrations can be carried out only along certain composition paths. Two kinds of paths are of interest in the calculations for liquid Cu-Fe-S mattes. In the first place, the Gibbs-Duhem integrations are readily carried throughout the two-phase regions if complete composition and sulfur-activity data are obtained for the tie-lines in these regions. Thus, in principle, for metal-saturated mattes, activities of all components can be calculated from sulfur activities and tie-line compositions all the way across the Cu-Fe-S isotherms, Figs. 2 to 4, from the Cu-S side to the Fe-S side, traversing first the liquid matte-liquid metal miscibility gap and then the liquid matte-solid iron region. The second type of composition path available for Gibbs-Duhem integrations is in the single-phase liquid-matte region. Here the integrations must follow paths of constant Fe:Cu ratio; that is, paths of constant E_{Fe}. On the ternary isotherms, Figs. 2 to 4, these paths are represented by straight lines radiating from the sulfur corner of the diagram.

By combining the two kinds of paths described above, it is theoretically possible to calculate complete activity data for all components throughout the entire liquid matte field, based on experimental measurements of sulfur activity and solution compositions. However, the present investigation did not cover the liquid matte-solid iron field. Hence, only at 1350°C, for which temperature the two-liquid miscibility gap extends well across the diagram, are sufficient data available to evaluate activities over a substantial portion of the matte region.

Miscibility Gap Integration at 1350°C—For a two-phase region in a ternary system, the activity of each component (a_1, a_2, or a_3) is the same in both phases, and two Gibbs-Duhem equations can be written governing activity changes throughout the two-phase region

$$n_1\, d \ln a_1 + n_2\, d \ln a_2 + n_3\, d \ln a_3 = 0 \qquad [1]$$

$$n_1'\, d \ln a_1 + n_2'\, d \ln a_2 + n_3'\, d \ln a_3 = 0. \qquad [2]$$

In these equations, n_i represents mols of component i in one phase while n_i' represents mols of component i in the other phase (at the other end of a tie-line). Now, by simultaneous solution of Eqs. 1 and 2, $d \ln a_3$ can be eliminated to obtain another useful Gibbs-Duhem relation

$$d \ln a_2 = -\frac{\left(\frac{n_1}{n_3} - \frac{n_1'}{n_3'}\right)}{\left(\frac{n_2}{n_3} - \frac{n_2'}{n_3'}\right)} d \ln a_1. \qquad [3]$$

By integration of Eq. 3, a_2 can be calculated from known values of compositions and a_1 for the tie-lines in a two-phase region. Activities of the third component (a_3) can be found by the same procedure, starting with elimination of $d \ln a_2$ between Eqs. 1 and 2.

For the miscibility gap in the Cu-Fe-S system, it is convenient to choose sulfur, Cu_2S, and FeS as the three components. Then Eq. 3 can be rewritten in

terms of the equivalent ratios E_{Fe} and E_s as follows

$$d \log a_{Cu_2S} = \left(\frac{E_{Fe}' - E_{Fe} + E_s' E_{Fe} - E_s E_{Fe}'}{E_{Fe}' - E_{Fe}} \right) d \log a_s \quad [4]$$

Owing to an interesting singularity of the Cu-Fe-S system, this equation proved difficult to integrate through part of the miscibility gap. That is, at 1350°C the tie-lines change considerably in direction along the miscibility gap, so that part way along a point is reached where the tie-line if extended would pass exactly through the sulfur corner of the composition triangle, see Fig. 4. At this point, the Fe:Cu ratio is the same in both phases and $E_{Fe} = E_{Fe}' = 0.5$. For this condition, the denominator of the right-hand member of Eq. 4 becomes zero. According to Eq. 4, at this same point $\frac{\partial \ln a_s}{\partial \ln a_{Cu_2S}}$ must also be zero, indicating a maximum or minimum in the H_2S:H_2 ratio. The fact that the experimental H_2S:H_2 ratios actually show a minimum very close to $E_{Fe} = E_{Fe}' = 0.5$, see Fig. 6, thus is a reassuring confirmation of the accuracy of the experimental work.

Further study of the type of singularity just pointed out led to the following generalization: *When an extension of a tie-line passes through the corner corresponding to one of the components, the isoactivity curves for that component in the adjacent single-phase fields must be tangent to the boundary curve of the immiscibility region at the ends of the tie-line.*

After trial of a variety of mathematical procedures, a reasonably satisfactory integration of Eq. 4 along the miscibility gap was finally accomplished by an iterative method of successive approximations. This method is based on the fact that graphical integration of a_{Cu_2S} vs composition data to find a_s is straightforward through the entire region, according to the following modification of Eq. 4

$$\Delta \log (p_{H_2S}/p_{H_2}) = \int \left(\frac{E_{Fe}' - E_{Fe}}{E_{Fe}' - E_{Fe} + E_s' E_{Fe} - E_s E_{Fe}'} \right) d \log a_{Cu_2S} \quad [5]$$

The fraction within the integral goes to zero when $E_{Fe}' = E_{Fe}$ and thus causes no difficulty. Accordingly, as a starting point, a simple trial relation of a_{Cu_2S} to composition was assumed; namely, that $d \log a_{Cu_2S} = d \log (1 - E_{Fe})$. Using this assumed relation, Eq. 5 was integrated to find a tentative relation of log (p_{H_2S}/p_{H_2}) to composition. Subtraction of these tentative values of log (p_{H_2S}/p_{H_2}) from the actual experimental values of log (p_{H_2S}/p_{H_2}) gave a set of *S-corrections* ($\delta \log p_{H_2S}/p_{H_2}$). These *S-corrections* were integrated according to Eq. 4 to obtain a set of *Cu,S-corrections* to be applied to the originally assumed values of log a_{Cu_2S}. By repetition of this procedure, a relation of a_{Cu_2S} was found which, when integrated according to Eq. 5, gave back the experimental values of log (p_{H_2S}/p_{H_2}) within ±0.002. The values of a_{Cu_2S} were adjusted to a standard state of pure liquid Cu_2S of exactly stoichiometric composition, using Schuhmann and Moles' data, and the results are plotted as curve *A* in Fig. 7.

To establish a reference state for a_{FeS}, the following chemical equation and its equilibrium constant were used

$$Fe(\gamma) + H_2S = FeS(liq) + H_2$$
$$k_{FeS} = \frac{(a_{FeS})}{(a_{Fe(\gamma)})(p_{H_2S}/p_{H_2})} \quad [6]$$

For the equilibrium of gas-liquid matte-liquid metal-solid iron at 1350°C, p_{H_2S}/p_{H_2} found experimentally was 4.44×10^{-4} (from Table I) and $a_{Fe(\gamma)}$ was estimated to be 0.93, based on examination of the Fe-Cu equilibrium diagram.[11] The value of k_{FeS} was estimated as 90 at 1350°C, based on Kelley's data.[12] Accordingly, a_{FeS} was estimated to be 0.371 for the terminus of the miscibility gap at the liquid matte-liquid metal-solid iron region. Now, with p_{H_2S}/p_{H_2} and a_{Cu_2S} already known as a function of matte composition along the miscibility gap, an ordinary ternary Gibbs-Duhem integration gave a_{FeS} as a function of matte composition along the miscibility gap. The results are plotted as curve *B* in Fig. 7.

Since the values of a_{FeS} were referred to a standard state of pure liquid FeS by using an estimated value of k_{FeS}, it should be kept in mind that all values of a_{FeS} might be in error by a constant factor equal to the ratio of the value of k_{FeS} used in the calculations to the true value of k_{FeS}.

With complete data on p_{H_2S}/p_{H_2}, a_{Cu_2S}, and a_{FeS} now available along the miscibility gap, the further calculations of a_{Cu} and a_{Fe} are relatively straightforward. These calculations are outlined in the subsequent paragraphs and are related to the compositions of the liquid-metal side of the miscibility gap.

Recent studies of the dilute solution of sulfur in liquid copper[13] indicated that these solutions do not deviate greatly from Raoult's law, so that a_{Cu} referred to pure liquid copper as a standard state may be taken as equal to the mol fraction. Thus, taking a value of 2 wt pct S for the sulfur solubility at 1350°C (after Smith[13]), a_{Cu} is estimated to be 0.96 at the high-copper, zero-iron end of the miscibility gap. Combining this with p_{H_2S}/p_{H_2} and a_{Cu_2S} already evaluated at this point, the following equilibrium constant is calculated

$$2\,Cu\,(liq) + H_2S = Cu_2S\,(liq) + H_2$$
$$k_{Cu_2S} = \frac{(a_{Cu_2S})}{(a_{Cu})^2 (p_{H_2S}/p_{H_2})} = 163 \text{ at } 1350°C. \quad [7]$$

With k_{Cu_2S} available, a_{Cu} was then calculated from p_{H_2S}/p_{H_2} and a_{Cu_2S} along the miscibility gap. In Fig. 8, the resulting values of a_{Cu} are plotted against mol fraction iron in the liquid-metal phase, this mol fraction being referred to 1 mol Cu + Fe as a basis.

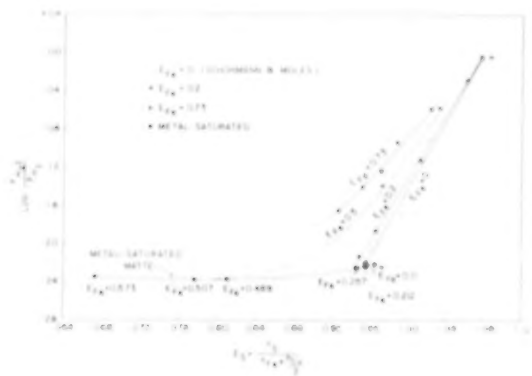

Fig. 6—Effect of matte composition on sulfur activity at 1350°C.

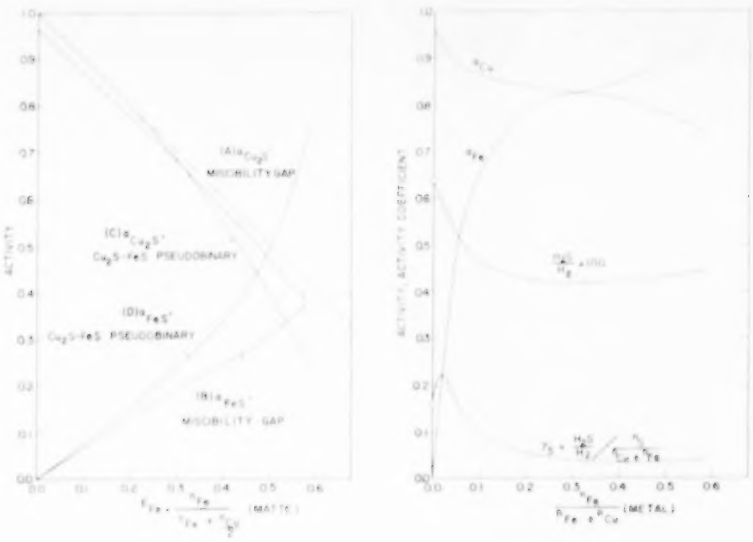

Fig. 7—LEFT: Activities of Cu_2S and FeS at 1350°C. Fig. 8—RIGHT: Activities of sulfur, copper, and iron in matte-saturated liquid metal at 1350°C.

The curve of a_{Cu} vs mol fraction is characterized by large positive deviation from ideality, approaching the condition of widespread immiscibility. As a matter of fact, this curve is very similar to the curve calculated by Chipman[11] for a_{Cu} vs mol fraction in the absence of sulfur but at a higher temperature (1600°C).

All the information necessary to calculate a_{Fe} along the miscibility gap at 1350°C has already been presented, including p_{H_2S}/p_{H_2}, a_{FeS}, and the value of k_{FeS}. Since the value of k_{FeS} was already used once in establishing a standard state for FeS, it cancels out in the overall calculations of a_{Fe}, so that the actual reference state for a_{Fe} is given by the previous choice of $a_{Fe} = 0.93$ for the three-phase region at 1350°C. Results of the calculations of a_{Fe} are plotted in Fig. 8. Here again, the strong positive deviation from ideality of the Cu-Fe solution is evident, with the activity coefficient of iron approaching about 10 in dilute solutions (ignoring the presence of sulfur).

Fig. 8 also shows how sulfur activity varies with composition of the matte-saturated liquid metal. The lowest curve gives an *activity coefficient* for sulfur, and it is of interest to note that this coefficient first increases slightly, then decreases markedly as iron is added to the liquid copper. However, the initial increase in γ_S is somewhat questionable, as it is calculated partly from results of other investigations of the Cu-S binary system[5,12] and partly from results of the present investigation. Clearly, a detailed investigation of the thermodynamic properties of the ternary liquid-metal solutions of sulfur and iron in copper should be of considerable interest in relation to the study of interactions of solutes in metallic solutions.

Matte-Field Integrations at 1350°C—Once complete activity data for all components were available along the miscibility gap, Gibbs-Duhem integrations could then be carried into the single-phase matte field along lines of constant E_{Fe} (that is, constant n_{Fe}/n_{Cu} lines, passing through the sulfur corner of the ternary isotherm). Taking sulfur, Cu_2S, and FeS as components, the following forms of Gibbs-Duhem relations were used[13]

$$\left(\frac{\partial \log a_{Cu_2S}}{\partial \log \frac{p_{H_2S}}{p_{H_2}}}\right)_{E_{Fe}} = \left(\frac{\partial n_S}{\partial n_{Cu_2S}}\right)\frac{H_2S}{H_2} \quad [8]$$

$$\left(\frac{\partial \log a_{FeS}}{\partial \log \frac{p_{H_2S}}{p_{H_2}}}\right)_{E_{Fe}} = \left(\frac{\partial n_S}{\partial n_{FeS}}\right)\frac{H_2S}{H_2} \quad [9]$$

The values of the partial derivatives at the right of Eqs. 8 and 9 were calculated from the intercepts of tangents to the sulfur-isoactivity curves on the S-Cu and S-Fe sides, respectively, of the ternary diagram.

Fig. 9 was found to be an especially convenient way of depicting the ternary isotherm and a large graph of this kind was actually used as the working diagram in carrying out the Gibbs-Duhem calculations. After laying out the various phase regions on the basis of the available data, the sulfur-isoactivity curves were located, using the smoothed data in Fig. 6. The sulfur-isoactivity curves are quite flat in this type of plot, and their two tangent intercepts at any point were easily determined at the two vertical axes at the left and right, respectively, of the diagram. Activities of Cu_2S and FeS were calculated along $E_{Fe} = 0$, 0.22, 0.4, 0.5, and 0.573, each calculation starting from the miscibility gap as a lower limit of integration. With these sets of values of a_{Cu_2S} and a_{FeS} established, the location of the isoactivity curves for Cu_2S and FeS, which are also plotted in Fig. 9, became a routine matter.

From the results of the calculations outlined above, the values of a_{Cu_2S} and a_{FeS} for the pseudobinary Cu_2S-FeS system were read off, and these are shown in curves *C* and *D* in Fig. 7. Curve *C* indicates that the pseudobinary solutions of Cu_2S and FeS exhibit moderate negative deviations from ideality. The shape of curve *D* is, of course, also consistent with this interpretation, but the standard state for FeS is somewhat uncertain, since it is based on an estimated equilibrium constant as discussed earlier. However, in making thermodynamic calculations based on these activity data, it will be helpful to keep in mind that these values of a_{FeS} are consistent with Kelley's estimate of the free energy of formation of liquid FeS.

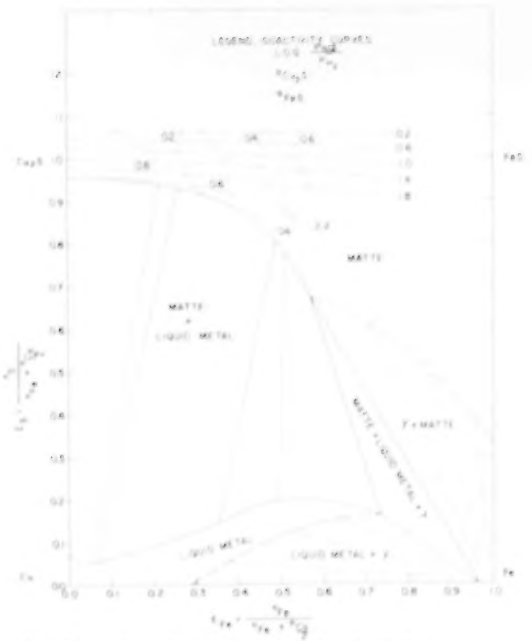

Fig. 9—Isothermal phase diagram for Cu-Fe-S system at 1350°C.

Comparison of the various activity curves in Fig. 7 shows that the assumption that ordinary copper mattes are ideal solutions of Cu_2S and FeS is a reasonable rough approximation for practical calculations. Such an approximation is particularly justified when accurate data on the sulfur deficiency are not available and there is an absence of information on the effects of oxygen.

Summary

Sulfur activities were measured experimentally as a function of phase compositions in three major regions of the Cu-Fe-S system at temperatures from 1150° to 1350°C. The three regions studied were a) liquid mattes, b) the liquid matte-liquid metal miscibility gap, and c) the γ-iron alloy-liquid matte-liquid metal region. A variety of Gibbs-Duhem integrations were used to calculate activities of Cu_2S, FeS, copper, and iron at 1350°C from the experimental data relating sulfur activity to composition.

The experimental data generally confirm previously published phase diagrams for the Cu-Fe-S system in the temperature range from 1150° to 1350°C and establish more accurately the locations of the liquid metal-liquid matte immiscibility region and the terminus of this region with two liquids in equilibrium with the γ-iron alloy phase.

The sulfur activities of liquid mattes are determined primarily by the ratio of equivalents of sulfur to total equivalents of copper plus iron, and are very sensitive to this ratio, the equilibrium $H_2S:H_2$ ratio changing as much as a hundredfold (one hundred and fourfold change in p_{S_2}) for only a 10 pct change in the sulfur:metal equivalent ratio. On the other hand, when the sulfur:metal equivalent ratio is held constant, the sulfur activity is found to be rather insensitive to the relative proportions of copper and iron in the matte. Similarly, the heat of solution of sulfur decreases markedly with decreasing sulfur:metal equivalent ratio, but is rather insensitive to Cu:Fe ratio.

As matte and metal compositions are varied along the miscibility gap at 1350°C, the variation in sulfur activity is rather small, but the sulfur activity passes through a minimum when the ratio of equivalents of iron to equivalents of copper is unity in both phases. For this condition, the tie-line extension passes through the sulfur corner of the ternary isotherm. Also, this condition caused difficulties in the Gibbs-Duhem integration.

The relationships of Cu_2S and FeS activities to matte composition, calculated by Gibbs-Duhem integrations, show that for approximate practical calculations, copper mattes might be considered as ideal solutions of Cu_2S and FeS. However, the FeS-Cu_2S pseudobinary solutions do show moderate negative deviations from ideality, see Fig. 7.

In the matte-saturated liquid-metal solutions at 1350°C, the activities of copper and iron show strong positive deviations from ideality, corresponding to the large tendency toward immiscibility of copper and iron at this temperature. However, the presence of sulfur markedly increases the solubility of iron in liquid copper at this temperature, as compared to the solubility in the absence of sulfur.

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Micrographic Study of Sulfide Roasting

The calcines resulting from the partial roasting of closely sized sulfide particles were examined by micrographic, X-ray diffraction, and chemical methods. The changes in composition of residual sulfide kernels show that the roasting process involves the preferential oxidation of metal atoms.

by P. G. Thornhill and L. M. Pidgeon

ALTHOUGH a considerable number of experimental investigations dealing with the roasting of sulfide minerals have been reported in the past,[1-10] the behavior of the single roasting particle does not appear to have received the attention it perhaps deserves. This is understandable in that the natural sulfide counterpart of the slab or sheet of metal normally used in high temperature oxidation studies is difficult to obtain and prepare, and is, in most cases, virtually impossible to maintain in one piece at roasting temperatures. The employment, on the other hand, of static aggregations of small particles in masses great enough to permit evaluation of the roasting reactions by means of thermobalances or gas analyses introduces other complications, such as localized overheating, sintering, and variable gas-solid contact.

In this investigation a compromise between the above extremes was attempted. Closely sized (e.g., 30 to 40 mesh) particles of natural sulfide minerals were isothermally roasted, out of contact with one another, in an air-swept system. The progress of the roasting reactions was followed by examination in polished section of the treated particles and by X-ray and chemical analysis of their residual sulfide kernels. In this way some conclusions respecting the mechanism of roasting reactions were drawn.

Roasting Furnace—The vertical tube furnace used for roasting the sulfide particles had a heated zone 10½ in. long in which four retractable light gage stainless steel trays were suspended. Each tray, and an accompanying baffle, was mounted on a 2 in. length of ⅜ in. stainless steel tubing. These assemblies were axially suspended on a 30 in. length of ¼ in. stainless tubing, by means of which they could be lifted from the hot zone to a water-cooled brass chamber at the top of the furnace. Since each tray unit could slide freely on the 30 in. tube, any desired number of the trays could be retained in the cooling chamber by the manipulation of a horizontal plunger during a momentary withdrawal of the four trays from the hot zone.

Although three heating coils, each equipped with variable external resistance, were employed, it was found impossible to avoid a drop of 10° to 15°C between the temperature at the top tray and that of the lower three. Consequently, only the bottom three trays were used for roasting. Temperature readings inside the furnace were made by means of a thermocouple inserted down the tray support tube at tray levels, and a separate thermocouple, inserted adjacent to the middle heating coil, was connected to a Micromax temperature control unit.

Experimental Method—The sulfide minerals were obtained in a form as free as possible from gangue and contaminating sulfides, and were crushed and screened to the required particle size range. The tray assembly was kept inside the furnace during the heating-up period and, when a steady temperature had been reached, the trays were withdrawn. Each tray was sprinkled with from 200 to 500 mg of the sulfide grains, and the assembly was lowered into the furnace without delay in order to minimize the time required to achieve the operating temperature. The furnace top was then clamped into position and the thermocouple inserted down the center tube to the required level. It was found that, when this procedure was used, the trays achieved their former temperature within 2 min, after which time the flow of dry air was begun at a rate of 1 liter per min, and timing of the run was started. At the required intervals of time the trays were successively withdrawn from the hot zone and stored in the cooling chamber by means of the retention plunger. On completion of the run the treated particles were removed from the trays to be set aside for examination.

The roasted particles were mounted in Lucite on a Buehler press operating at a temperature of 140°C

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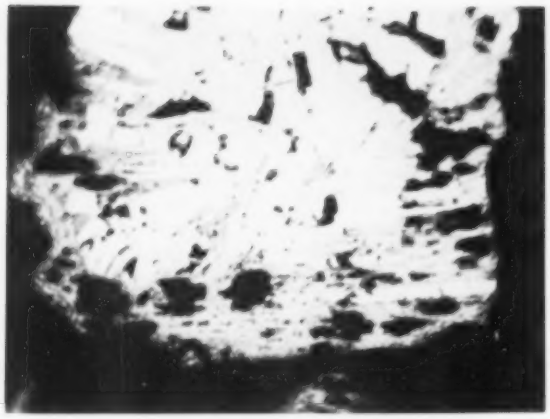

Fig. 1—65 to 80 mesh pyrrhotite, oxidized 17 min at 550°C. X500. Reduced approximately 10 pct for reproduction.

and at the lowest possible pressure, in order to avoid distortion of the friable oxide coatings surrounding the sulfide kernels. It was found that several samples, representing up to six successive stages of treatment, could be mounted in one plastic cylinder when short separatory wires were arranged spokewise on the bottom of the mold. Each sector could be made to contain a score or so of 30 to 40 mesh particles. The specimens were then gently taken down on the customary succession of emery papers until it was estimated that the particles had been center sectioned. Polishing was done on a fairly fast wheel covered by a well worn selvyt cloth treated with an aqueous slurry of *Linde A* brand alumina.

A Tukon microscope was used in some cases to measure the thickness of regular oxide coatings. Actual measurement at high power was preceded by preliminary scanning of a specimen at low power in order to arrive at a region in the specimen which was judged to be representative of the large number of grains comprising the specimen. Qualitative observations and micrographs were made with a Bausch & Lomb metallograph.

It was found that, by the use of hydrochloric acid at the proper strength and temperature, the sulfide kernels in partially roasted sulfide particles could be freed of their oxide coatings by selective leaching. The clean residual sulfides were then subjected to X-ray diffraction and, in some cases, chemical analysis.

Results of the Investigation

Simple Sulfides—*Sphalerite:* Sphalerite particles were found to oxidize at measurable rates over a relatively wide range of temperature, with the formation of quite uniform oxide coatings. Since the particles underwent no apparent change in shape or dimension during treatment, direct measurement of oxide thickness, and hence of oxidation rate, was possible, and the linearity of the rates permits their tabulation, as shown in Table I.

Each of the rates in Table I was taken from six-point curves obtained over periods ranging from 40 min at 750° to over 200 min at 660°C. The observed absorption of mounting plastic by the oxide jackets indicated a fine, regular porosity of the oxide. Debye-Scherrer patterns made from the residual sulfides were identical with those of the raw sphalerite, and there was no visual evidence of structural change in the kernels. The linearity of the roasting rates is a good indication that, under the conditions employed, the rate of reaction at the sulfide-oxide interface is the rate controlling mechanism, and gaseous diffusion processes through the oxide coating do not limit the overall reaction rate. This is supported by the relatively low calculated value of the Pilling and Bedworth ratio[11] and the visual evidence of regular porosity in the oxide.

Pyrite: Unlike sphalerite, both 30 to 40 and 65 to 80 mesh pyrite grains reacted at measurable rates over an extremely limited temperature range. The rate of advance of the oxide-sulfide interface at a furnace temperature of 492°C, for example, was estimated to be about 2.3 μ per min, while at a furnace temperature of 500°C, 30 to 40 mesh particles were dead roasted within 10 min, indicating a rate in excess of 21 μ per min. On the assumption of black body conditions and that the heat generated in the roasting particles is lost mainly by radiation, temperature gradients between the particles and their surroundings of 20° and 200°C respectively were calculated for the above two rates, so that the particles roasted at a furnace temperature of 500° probably approached 700°C by self-heating. Such an increase is sufficient to bring the pyrite well within the range in which it exerts sulfur vapor pressures of sufficient magnitude to affect markedly the reaction rate.

There was no visual evidence of crystallographic change in the residual sulfide kernels, and X-ray powder patterns of the latter were identical with those of the original mineral.

Table I. Roasting of Sphalerite

Mesh	Theoretical Average Particle Diam	Temperature, °C	Oxidation Rate, μ per Min
30 to 40	500 μ	540	nil
		680	0.16
		700	0.4
		725	1.2
		750	1.8
65 to 80	190 μ	605	nil
		660	0.17
		700	0.5

Pyrrhotite ($Fe_{1-x}S$) and Troilite (FeS): Pyrite did not appear to be suitable for this type of investigation because of its tendency to ignite in the air atmosphere employed in the furnace. Pyrrhotite, on the other hand, roasted at measurable rates over a relatively wide temperature range. While 65 to 80 mesh particles of this mineral roasted linearly at 550°C, the oxidation rate of the 30 to 40 mesh particles was retarded after the formation of about 160 μ of oxide. The oxidation rates, in the linear parts of the rate curves, are given in Table II.

Fig. 1 illustrates the columnar reaction interface obtained in partially roasted pyrrhotite grains. Examination of the partly oxidized polycrystalline particles under crossed Nichols revealed that changes in orientation of the oxide columns occurred only at intercrystalline boundaries which involved changes in orientation of the pyrrhotite, confirming the supposition that the pyrrhotite oxidized preferentially along certain crystallographic planes. According to Seitz,[11] atomic diffusion rates in anisotropic metals are extremely dependent upon the relation between the crystallographic orientation and the direction in which diffusion occurs. This suggests that ionic dif-

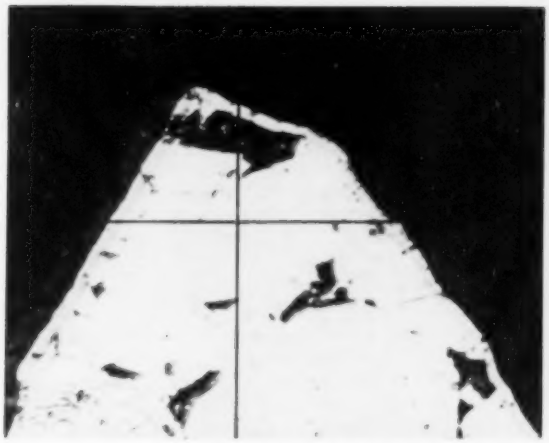

Fig. 2—30 to 40 mesh troilite (FeS), oxidized 35 min at 650°C. X360. Reduced approximately 20 pct for reproduction.

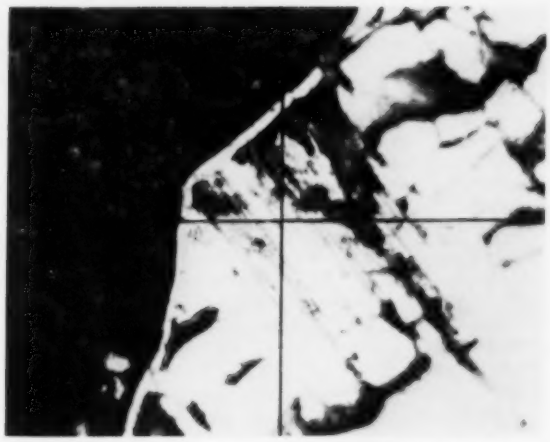

Fig. 3—30 to 40 mesh troilite (FeS), oxidized 60 min at 650°C. X360. Reduced approximately 20 pct for reproduction.

fusion at the reaction zone in solid sulfide is an important part of the mechanism of this particular roasting reaction. Wagner[10] has shown that the mobility of cationic species in a sulfide is much greater than that of the anions, so that it might be expected that, if diffusion in the sulfide plays a part in the roasting reactions, the metal component of the sulfide could be oxidized more rapidly than the sulfur, leaving a sulfide kernel having a higher sulfur to metal ratio than that of the original mineral. This could, of course, occur only in sulfides which can exist over a wide range of composition, and of which pyrrhotite is a good example.

In order to test the validity of the above considerations, a series of roasting tests were conducted on artificially prepared troilite (FeS). This material was made by reacting stoichiometric proportions of iron and sulfur powder in an evacuated glass bomb. The FeS sinter was fused, crushed, and sized to 30 to 40 mesh. Unlike pyrrhotite, this material was nonmagnetic, and X-ray diffraction determination of its lattice constants indicated that they were in good agreement with those obtained by Haraldsen[11] for FeS. The 30 to 40 mesh particles were roasted for periods up to 60 min at 550°, 600°, and 650°C. Examination of the calcines in polished section indicated that such oxidation as did occur in the 550° and 600°C specimens was manifested by a thin, dense layer of hematite on the surface of each particle. With temperature increased to 650°C, the coatings were thicker and contained a magnetite layer between the sulfide and hematite in the particles roasted for 35 min, as shown in Fig. 2. As can be seen in Fig. 3, however, a further 25 min treatment resulted in no increase in thickness of the dense layer, but rather in localized intergrowths of columnar oxide similar to those formed in the roasting of pyrrhotite.

The dense nature of the oxide coatings formed on the troilite, their evidently strong adhesion to the base sulfide, and the double layer of oxide representing two stages of iron oxidation presented an overall picture remarkably similar to the conditions which occur in the high temperature oxidation of metallic iron. The particles roasted at 650°C were leached with dilute hydrochloric acid to remove their oxide jackets, after which they were found to have become increasingly ferromagnetic with duration of roasting, indicating that the oxidation had indeed resulted in a decrease in Fe:S ratio in the sulfide and that it had become pyrrhotite. The extent of the initial selectivity of oxidation between the iron and sulfur was determined by interpolation of the data reported by Haraldsen[11] relating unit cell volumes of the troilite-pyrrhotite series with atomic pct sulfur. X-ray measurements in the present case were made with iron $k\alpha 1 = 1.932076\ kX$, and the lattice constants obtained are given in Table III.

The S:Fe ratios indicated in the last column of Table III show that the preferential oxidation of the iron had virtually ceased after 35 min, so that while it is apparent that the processes which occurred during this period involved the reaction

$$\text{FeS} + \text{oxygen} \rightarrow \text{Fe}_{1-x}\text{S} + 0.05\ \text{Fe}_2\text{O}_3 \qquad [1]$$

the reaction which took place during the remainder of the 60 min period was essentially

$$\text{Fe}_{1-x}\text{S} + \text{oxygen} \rightarrow 0.45\ \text{Fe}_2\text{O}_3 + \text{SO}_2. \qquad [2]$$

Using the calculated densities of the solids involved in Eq. 1, a simple calculation similar to those initiated by Pilling and Bedworth[12] shows that the volume of the oxide formed is greater than that lost by the sulfide by a factor of 2.66. The subsequent process represented by Eq. 2, on the other hand, can be shown in the same way to result in a volume of oxide amounting to no more than 77 pct of the space vacated by the reacted sulfide. The tendency for this sulfide to oxidize with the formation of the columnar intergrowths extending into the particle along preferred crystallographic directions is thus easily accommodated in process 2, with a possible 23 pct porosity in the oxide for the passage of gases to and from the reaction interface. Conversely, the high degree of net expansion which results from process 1 frustrates this tendency at the outset, forcing more than half of the oxide to grow outwardly from the datum line given by the original FeS surface. Under these conditions it is likely that the porosity of the oxide is nil, and that the mechanism of the reaction is similar to that obtaining in the high temperature oxidation of metallic iron, i.e., the transfer of electrons and cations through the oxide layer followed by their reaction with the oxygen at the outer oxide surface.

During the period that process 1 predominates, any crack which might develop in the oxide would, by oxide growth, be self-healing. As process 2 begins to predominate, with the onset of volumetric ratios less than unity, cracks in the oxide shell must remain as such, permitting the passage of gases necessary for the progress of the roasting reaction proper. This is evident from the relation between the thickness of the outer oxide skin and the depth of penetration of the columnar oxide shown in Fig. 3, in that the reactions forming the latter must have taken place at rates many times greater than that forming the outer skin, and hence took place by a different reaction mechanism, doubtless one which permitted the access of oxygen to the sulfide-oxide interface. In this connection it is to be noted that the dense outer layer of oxide as formed on the troilite was practically nonexistent in the case of the pyrrhotite roasts, and, as seen in Fig. 1, the pyrrhotite was deeply penetrated by oxide columns even though it was roasted for a shorter period and at a temperature 100° lower than that at which the troilite was treated.

Table II. Oxidation of Pyrrhotite

Mesh	Theoretical Average Particle Diam	Temperature, °C	Oxidation Rate, μ per Min
30 to 40	500 μ	502	0.14
		532	1.40
		550	2.0
65 to 80	190 μ	550	2.55

Chalcocite (Cu_2S): Both artificial and natural chalcocite particles were roasted in air for various periods at temperatures ranging from 420° to 550°C. Examination of the resulting calcines revealed that, as in the roasting of troilite, the primary oxidation product appeared as a dense layer in contact with the sulfide surface. Increased periods of treatment were found to have resulted in the growth of porous oxidation products in a secondary layer with little, if any, increase in the thickness of the dense outer layer. As in the case of troilite, the onset of the secondary reactions was marked by an increased rate of advance into the particle of the reaction interface, again implying a change in mechanism. In contrast to the troilite case, however, was the apparent expansion of the secondary layer during its growth, which caused the outer layer to break away from the particle.

The 30 to 40 mesh natural chalcocite particles, roasted at 492°C and leached with dilute acid for the selective removal of the oxidation products, were subjected to X-ray diffraction analysis. Debye-Scherrer films showed that the sulfide kernels were approximately 50 pct converted to digenite after a 7 min treatment and that the transition to digenite was virtually complete in 19 min. The conversion to digenite is another point of similarity between the behavior of troilite and chalcocite since the cubic digenite phase, like pyrrhotite, exists as a defect structure, which permits considerable deviation from the stoichiometry of the orthorhombic chalcocite. The possible extent of this deviation was shown by Buerger[10] to result in deficiencies of copper equivalent to the formula $Cu_{1.8}S$ at a temperature of 300°C. The decreasing Cu:S ratio in the digenite series parallels a shrinkage of the lattice constant, and while the cell edge of a specimen of natural digenite filmed for comparative purposes was found to be 5.57 *kX* units, those of the roasted chalcocite were 5.52 and 5.49 *kX* units for the 7 and 19 min calcines, respectively. This indicates that the copper had been depleted to an extent even greater than that exhibited by natural digenite ($Cu_{1.8}S$). However, in the absence of other data, the calculations of the volumetric ratios were based on the reactions

$$Cu_2S + \text{oxygen} \rightarrow Cu_{1.8}S + 0.1\,Cu_2O \qquad [3]$$

and

$$Cu_{1.8}S + \text{oxygen} \rightarrow 0.9\,Cu_2O + SO_2. \qquad [4]$$

The volumetric ratio for Eq. 3 is 1.94; that for Eq. 4 would be 0.79. Subsequent reaction of the Cu_2O with O_2 and SO_2 to form, for example, $CuSO_4$ could increase the latter ratio to about 3.7, accounting for the expansion which was observed in the inner layer.

Complex Sulfides—*Chalcopyrite*: The 30 to 40 mesh chalcopyrite particles, treated in air at 550°C, were found to roast with the formation of large gaps, or holes, in the oxidation products. As seen in Fig. 5, the latter did not appear as a continuous layer with the regular porosity exhibited by the sphalerite coatings but, rather, as one or more layers of dense appearance enclosing the gaps. The relative thinness of the outer layers indicates that the formation of the gaps took place early in the treatment, and the solid and regular nature of the oxide growth which ensued suggests a change in mechanism.

Table III. Roasting of 30 to 40 Mesh Particles of FeS at 650°C

No.	Time, Min	*a kX*	*c kX*	*c/a*	*V kX*³	Atomic Pct S*
FeS	0	3.438	5.862	1.705	60.00	50.00
7	15	3.438	5.749	1.672	58.84	52.08
8	35	3.433	5.718	1.665	58.36	52.72
9	60	3.433	5.714	1.664	58.32	52.78

* Interpolated from Haraldsen's data.

Definite phase changes in the sulfide were seen to parallel the growth of the oxidation products. The first was a Widmanstaetten transformation manifested by the appearance of an isotropic brownish purple phase with the brassy yellow chalcopyrite. The new phase, as shown in Fig. 4, consolidated initially at the oxidation interface, whence it grew inwardly, until the entire sulfide kernel was transformed. Continued roasting resulted in the change in color of this phase from brownish purple through mauve to pale blue, after which a second Widmanstaetten transformation involving another blue, but anisotropic, phase occurred.

Debye-Scherrer powder patterns were made from some of the calcines resulting from the 550°C roasting series after removal of the oxides by HCl leaching, and the extent to which preferential oxidation of iron had taken place was determined by chemical analysis. The changes taking place in the sulfide kernels are summarized in Table IV.

The chemical analyses in Table IV are accurate in respect to the Cu-Fe relationship, but only approximate in absolute value with respect to the actual sulfide, since the original particles were contaminated with gangue minerals. Measurement with any accuracy of the angular spacings for the digenite phase was only possible in the case of the powder

pattern resulting from the 20 min sulfide because of the interference of either chalcopyrite or covellite lines in the other three patterns. The small unit cell dimension of the digenite phase, while due in part to the presence in the lattice of iron atoms, is indicative of a low metal:sulfur ratio and, consequently, of a high proportion of lattice vacancies. While there is no guarantee that the digenite structure existed as such at 550°C, it can be safely assumed that the phase at this temperature was no less disordered than the digenite at room temperature, and hence provided a medium favorable to diffusion. The color changes of the digenite phase which were noted earlier as having taken place with the progress of the roast can be inferred from Table IV to have coincided with its depletion in iron.

The mechanism of the chalcopyrite roast at 550°C appears to be one in which the digenite phase plays a key part. Once formed by the initial burning of sulfur and iron at the surface of the chalcopyrite particle, this phase permits the rapid diffusion, inwardly of copper and outwardly of iron, which is necessary to account for the selectivity of oxidation indicated in Table IV. The digenite-chalcopyrite interfaces advance with continued replacement of iron by copper until the entire kernel is converted to the digenite phase. Since the rate of advance of these interfaces is very much more rapid than that of the oxide-sulfide interfaces, it appears likely that the resulting rapid rate of shrinkage involved in the chalcopyrite-digenite conversion is a contributing factor in the formation of the double or multilayered structure of the solid oxidation products. Thus, just as the period of this conversion was found to coincide with the formation of the multilayered oxide structure, its completion was marked by the beginning of growth of the innermost oxide layer, which showed no further tendency to separate from the sulfide for the remainder of the roast. The increasingly protective nature of the inner layer retards the oxidation reaction, apparently to the extent that the copper roasts in preference to the sulfur, its concentration in the kernel decreasing to the point at which the digenite begins to transform to covellite. The oxide formed in the later stages of the roast is shown in Fig. 6.

Table IV. Roasting of 30 to 40 Mesh Chalcopyrite at 550°C

Time, Min	Sulfide Analysis, Pct Cu	Fe	Phases Present in Sulfide
nil	35.3	30.6	Chalcopyrite
12	—	—	Brownish purple digenite, chalcopyrite
20	60.6	8.3	Mauve digenite, a_o = 5.36 kX
35	68.0	nil	Blue digenite, covellite
50	68.0	nil	Blue digenite, covellite

Bornite (Cu_5FeS_4): A relatively high temperature (660°C) was found necessary to effect the roasting of 30 to 40 mesh bornite particles at a rate comparable to that obtained in chalcopyrite at 550°C. As in the chalcopyrite case, the roasting was characterized by the early conversion of the bornite to a digenite phase (a_o = 5.51 kX), changing the pink bornite color through mauve to blue. Continued roasting resulted in the development of a Widmanstaetten structure optically identical with that shown in the chalcopyrite roast to attend the conversion of the digenite phase to covellite.

Localized zones of shrinkage, apparently conforming to certain crystallographic planes, and resulting in the formation of gaps between primary and secondary oxide layers, and in some cases between sulfide and oxide as shown in Fig. 7, were found to occur in all specimens. Table V summarizes the changes taking place in the sulfide kernels.

Again, accurate measurement of the digenite lattice constant was possible only in the single phase member of the series. The fact that the bornite unit cell can approximately accommodate eight digenite unit cells gives rise to a broadening of the Debye-Scherrer lines of the 6-min sulfide in accordance with the relationship shown in Table VI. The chalcopyrite roast showed a similar facility of transformation in that its unit cell comes close to accommodating two cells of the digenite phase to which it was converted.

Pentlandite ($NiFe)_9S_8$: Pentlandite was roasted in particle sizes ranging from 30 to 40 mesh to 100 to 200 mesh and at temperatures ranging from 550° to 725°C. At temperatures below 650°C, the oxidation was seen to proceed, in its initial stages, in a manner similar to that displayed by troilite, with the formation of firm, protective oxide shells. This process was accompanied by the conversion of the sulfide to a polycrystalline anisotropic phase which was retained throughout the roasting period at the lower temperatures but which, above 650°C, was converted in turn to two new sulfide phases. The higher temperature roasts and the later stages of the lower temperature roasts were marked by the formation of shrinkage gaps as in bornite and chalcopyrite.

X-ray diffraction analysis on some of the sulfide kernels showed that the first conversion mentioned above was from pentlandite to a phase isomorphous with pyrrhotite. The lattice constants of the pyrrhotite phase are given in Table VII. Radiation: iron $k\alpha 1$ = 1.932076 kX.

Analysis of the oxide shells obtained in the 67 min sample permitted calculation of a metal balance which indicated that, while the oxide contained 52 pct of the iron originally in the pentlandite, only 8 pct of the total nickel had been oxidized. This was in reasonably good agreement with the average thickness of the oxide shells, 7.2 μ, surrounding the 190 μ particles.

It is apparent from the data in Table VII that the mechanism of the pentlandite roast is analogous to that of troilite, since the conversion of the pentlandite to the pyrrhotite form necessarily involves the initial preferential oxidation of the metals. Since it is known[6] that the high nickel forms of the pyrrhotite phase hold much more closely to the stoichiometric metal:sulfur ratio than do the true pyrrhotites, the shrinkage in unit cell volume reported in Table VII is probably due mainly to the increasing proportion of nickel atoms substituting for the larger iron atoms in the lattice.

Table V. Roasting of 30 to 40 Mesh Bornite at 660°C

Time, Min	Sulfide Analysis, Pct Cu	Fe	Phases Present in Sulfide
nil	63.36	11.14	Bornite, a_o = 10.93 kX
6	67.75	9.36	Bornite, mauve digenite
18	72.73	0.75	Blue digenite, a_o = 5.51 kX
47	73.70	nil	Blue digenite, covellite

At a temperature of 685°C, 80 to 100 mesh pentlandite particles were found to undergo a second

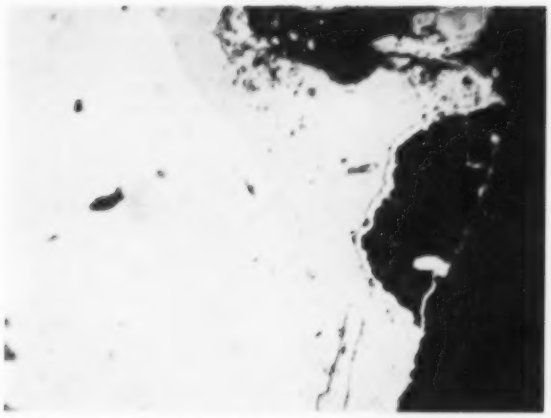

Fig. 4—30 to 40 mesh chalcopyrite, oxidized 6 min at 550°C. X500. Reduced approximately 15 pct for reproduction.

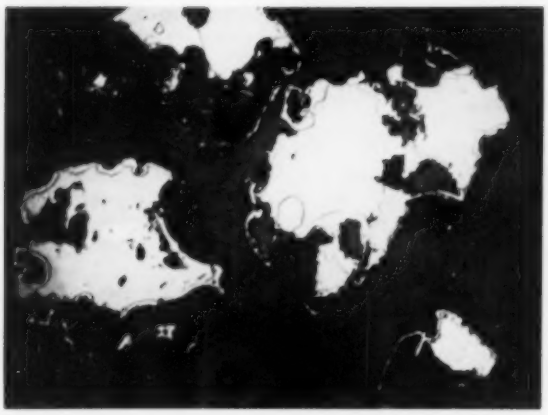

Fig. 5—30 to 40 mesh chalcopyrite, oxidized 12 min at 550°C. X75. Reduced approximately 15 pct for reproduction.

phase change after elimination of the iron by selective oxidation. The sulfide kernels of these particles roasted 30 min analyzed 67.3 pct Ni and 2.4 pct Fe. The sulfide phases were identified by X-ray diffraction to be chiefly Ni_3S_2 and a phase conforming to the data given by Lundqvist[7] for Ni_7S_6, with a relatively small proportion of the pyrrhotite phase. It is thus evident that the inward diffusion of nickel, initiated during the selective oxidation of the pentlandite and pyrrhotite phases, continued at the higher temperature even after virtual elimination of the iron, resulting in the preferential oxidation of sulfur at the reaction interface. The shrinkage of the sulfide kernel which accompanies this process may easily account for the exaggerated gap formation which was observed to have taken place.

Table VI. Effect of Bornite-Digenite Conversion on Debye-Scherrer Lines

Bornite, *hkl*	6 Min Roast, Bornite + Digenite		18 Min Roast, *hkl* Digenite
311	—• broadening	—•	111
222			
400	—• broadening	—•	200
331	—• disappearing		
440	—• broadening	—•	220
622	—• broadening	—•	311

Discussion

From the results of the present investigation, the case in which the sulfide kernel retains the composition and crystal structure of the original mineral after partial roasting appears to be the exception rather than the rule. Thus, the sulfide kernels occurring in partially roasted FeS, Cu_2S, chalcopyrite, bornite, and pentlandite particles were found to have converted, by the preferential oxidation of one or more of their components, to sulfide phases having defect structures, while sphalerite and pyrite were the only minerals in which such changes were not detected. (The pyrrhotite used in this experimental work probably also falls into the latter class, since in its case the defect structure obtained at the outset, although accurate lattice parameter measurements were not made on the partially roasted sulfide to determine whether the sulfide kernels had suffered further depletion in iron.) Determination of roasting rates by measurement of the thickness of oxide shells was successful only in the case of sphalerite and pyrrhotite, although the columnar nature of the sulfide-oxide interfaces obtaining in the pyrrhotite roast rendered accurate measurement difficult, if not impossible. Pyrite, the third member of this group, displayed so exaggerated a sensitivity to temperature in its roasting reactions that accurate measurement of roasting rates in air was impossible. On the other hand, kinetic studies on the roasting of those sulfides which undergo selective oxidation are understandably complicated by the fact that the oxidation takes place according to mechanisms which differ from one stage of a roast to the next, and the irregular nature of the multilayered oxidation products precluded any but the roughest estimation of oxidation rates.

The tendency toward preferential oxidation of the metallic components of the low sulfur minerals FeS, Cu_2S, and pentlandite and of the sulfur in chalcopyrite suggests that the oxidation of metal and sulfur does not take place simultaneously even after the conversion of the sulfide to its equilibrium defect structure. Taking the FeS roast as a specific example, it was found that the preliminary stages of oxidation had resulted in an overall reduction in the Fe:S ratio in the residual sulfide, apparently by diffusion of iron from the center to the surface of the kernel. The necessary concentration gradient implies a lower concentration of iron at the oxidation interface than at the center of the particle and, conversely, a relatively high concentration of sulfur at the surface. It is conceivable that the latter can increase to the point at which the surface sulfide exerts sufficient sulfur pressure to cause localized dis-

Table VII. Roasting of 65 to 80 Mesh Pentlandite at 600°C

Time, Min	Sulfide Analysis, Pct Ni	Fe	a kX	c kX	c/a	V kX^3	Phases Present
nil	35.1	32.3	10.01	—	—	—	Pentlandite
17	—	—	3.432	5.530	1.611	56.40	Pyrrhotite type
37	—	—	3.432	5.510	1.605	56.19	Pyrrhotite type
67	42.2	22.0	3.432	5.500	1.603	56.09	Pyrrhotite type

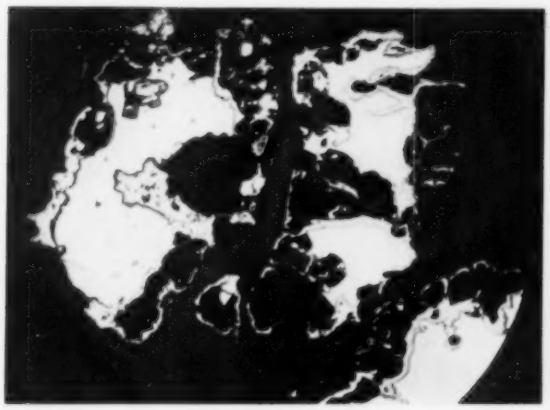

Fig. 6—30 to 40 mesh chalcopyrite, oxidized 40 min at 550°C. X75. Reduced approximately 15 pct for reproduction.

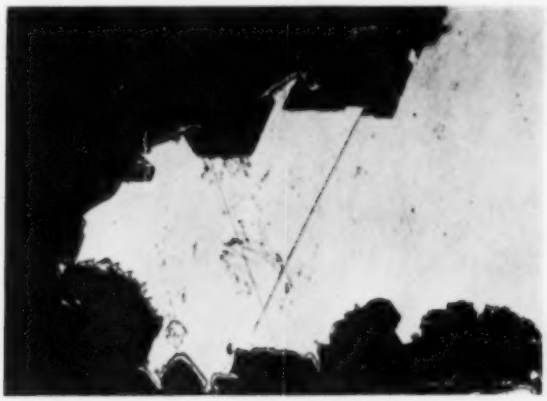

Fig. 7—30 to 40 mesh bornite, oxidized 36 min at 660°C. X150. Reduced approximately 15 pct for reproduction.

ruption of the oxide film, permitting the ingress of oxygen to combine with the sulfur and to begin forming a fresh iron oxide film. Growth of the new film by ionic and electronic transfer would suffer early interruption by again causing a local rise in sulfur concentration at its surface of contact with the sulfide. The continued repetition of such a process would result in the gas-permeable oxide layers which are observed to have formed during the second stage of the roast, when iron and sulfur oxidize at equal rates.

Unlike FeS and Cu_2S, chalcopyrite was seen to have been converted, early in the roast, to a phase having a higher metal:sulfur ratio than that of the original mineral, indicating that sulfur had initially oxidized at a greater rate than had the metals. It might be expected, then, that the metal oxide films formed initially would suffer more violent disruption by the higher ratio of released sulfur than obtained in the case of the lower sulfides. The visibly porous nature of the outer oxide layers formed on chalcopyrite indicated that this was indeed the case, and it is likely that the shrinkage gaps were, in many cases, enlarged by distention of the outer oxide layer. Large scale rupture of this layer would then be followed by a renewed growth of oxide on the exposed sulfide surface. This mechanism is supported by the noticeably thinner and, in some cases, nonexistent, layers of oxide on many of the inner surfaces of the shrinkage gaps occurring in the chalcopyrite calcines. In this connection, it is worth noting that both bornite and pentlandite roasted with the formation of oxide layers of such apparent density, and at such relatively low rates, as to suggest that much of the oxide growth took place by ionic migration through a continuous oxide layer. It seems probable that the bare sulfide surfaces seen in some of the shrinkage gaps formed in these minerals are localized sites of outward sulfur elimination by dissociation from the surface, and metal elimination by migration of the cations through the sulfide to a region of firm contact with the oxide layer, through which they diffuse to the air-oxide interface.

Summary

By micrographic, X-ray diffraction, and chemical methods of examining partially roasted particles, the majority of the sulfide minerals investigated were shown to display a marked selectivity in oxidation between one or more of their contained elements. This observed selectivity of oxidation has permitted certain conclusions to be drawn respecting the mechanisms of the roasting reactions in their initial stages, and has implied a mechanism whereby oxidation continues after it has ceased to be measurably selective. This same implied reaction mechanism, i.e., the growth of oxide films by cationic migration and their periodic disruption by built-up sulfur pressure at the sulfide-oxide interface, may well be applicable to the roasting of all sulfides, including sphalerite, which is known to hold rigidly to its stoichiometric composition, and hence did not undergo the measurable changes observed in the other minerals on roasting.

Acknowledgments

The authors gratefully acknowledge the financial assistance of the Consolidated Mining and Smelting Co. in supporting that part of the work done at the University of Toronto in 1950-1951. Special thanks are also due to Falconbridge Nickel Mines Ltd. for permission to publish the portion which was carried out in their laboratories, and to C. Law McCabe, Carnegie Institute of Technology, for helpful discussions.

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Discussion of this paper sent (2 copies) to AIME by Sept. 1, 1957 will appear in AIME *Transactions* Vol. 212, 1958, and in JOURNAL OF METALS, October 1958.

Zone Refining of Bismuth

by J. H. Wernick, K. E. Benson, and D. Dorsi

BISMUTH containing less than 10 ppm of metallic impurities has been prepared by zone refining material containing approximately 1000 ppm. By zone refining seven bismuth ingots of different initial purities, it was determined that silver, copper, lead, tin, nickel, magnesium, calcium, and iron are segregated. All except iron have distribution coefficients less than unity. Silver, copper, lead, and tin are not detected spectroscopically at the pure end. Nickel is concentrated at the impure end, even though it is not detected in the original bismuth. Twelve passes are all that are required for silver, lead, tin, and nickel.

The bismuth to be zone refined was first drip-melted at a pressure of approximately 0.025 mm Hg into a Pyrex tube as shown in Fig. 1. This drip-melting operation removes second phase impurities such as oxides. After drip-melting, the tube was sealed off under vacuum at the constriction. It was then placed in a larger tube, which was evacuated to a pressure of 0.025 mm Hg and sealed off. This larger tube conveniently served as a support for the ingot tube during zone refining.

Table I. Zone Refining Data for Bismuth*

Element	Original	Distance from Lead End in In.† 0	3	6	9	11
Spectrographic Qualitative Analysis						
Ag	1	—	—	—	—	1
Bi	7	7	7	7	7	7
Ca	—	1	1	1	1	—
Cu	1	1	1	1	1	2
Mg	1	1	1	1	1	1
Pb	2	—	—	—	—	3
Si	1	1	1	1	1	—
Quantitative Analysis‡						
Ag	—	—	—	—	—	0.0001
Cu	0.0002	0.0001	0.0001	0.0001	0.0001	0.004
Pb	0.001	—	—	—	—	0.02
Fe	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001

* Lot 6, 45 passes.
† Numbers refer to: 1, very slight trace, <0.001 pct; 2, slight trace, <0.005 pct; 3, trace, <0.01 pct; 7, principal, >10 pct; and dash, not detected.
‡ The elements Sb, Tl, Mg, Mn, Sn, Si, Cr, Ni, Al, Ca, In, Cd, and Zn were not detected in the original bismuth and zone refined ingot.

The zone refiner used for nearly all of the experiments was an induction heated automatically reciprocating unit[1] shown in Fig. 2. The starting position was as shown on the figure. The travel rate was 1.8 in. per hr in the forward direction and 1.8 in. per sec in the reverse direction, and the distance traveled was one third the length of the ingot. The boat was held at an angle of 3° to the horizontal to prevent mass transfer.[2]

J. H. WERNICK, Associate Member AIME, K. E. BENSON, and D. DORSI are associated with Bell Telephone Laboratories Inc., Murray Hill, N. J.

TN 414DE. Manuscript, Feb. 19, 1957.

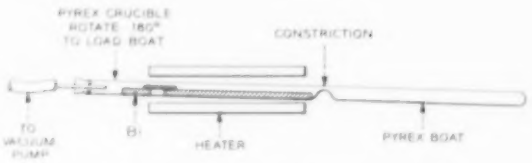

Fig. 1—Drip-melting system for loading boat.

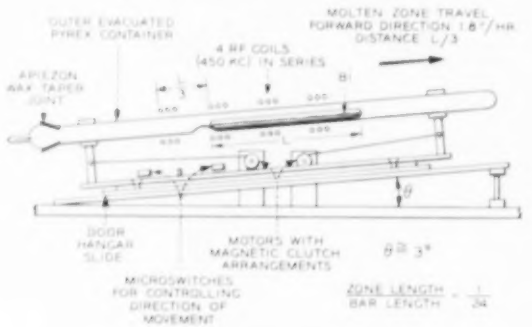

Fig. 2—Automatic reciprocating zone refiner.

The spectroscopic qualitative analytical data for one zone refined ingot are shown in Table I as an example of the results. A quantitative analysis of this same ingot was performed by J. S. Smart, Jr., of the American Smelting and Refining Co. Research Laboratory. These data are also shown in Table I.

Some work on analytical sample preparation indicates that the very slight traces of calcium and silicon reported in the qualitative results are probably due to pickup during etching of the analytical samples. The analytical samples used for the quantitative analysis were not etched. The analytical results show the absence of calcium and silicon, but show the presence of iron, which may be due to sampling. Bismuth intentionally doped with 0.03 pct Ca and Mg was zone refined, and these two elements were segregated.

Acknowledgments

The authors wish to thank W. G. Pfann for his continued help and interest throughout this work. The large contribution made by D. Babusci and other members of the Analytical Dept. is greatly appreciated. Thanks are due A. J. Phillips and J. S. Smart, Jr., of the American Smelting and Refining Co. Research Laboratories for the quantitative analysis and helpful discussions.

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AIME OFFICERS:

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PAST-PRESIDENT—C. E. REISTLE, JR.

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FIELD SECRETARY & ASS'T. SECY.—R. E. O'BRIEN, 707 NEWHOUSE BLDG., SALT LAKE CITY 1, UTAH

AIME NEWS

Three Divisions Nominate Officers To Serve in 1958

In The Metallurgical Society of AIME, the Nominating Committees of the three Divisions: Extractive Metallurgy, Institute of Metals, and Iron and Steel, have submitted the names of officers to serve in 1958.

Officers nominated to serve EMD in 1958 are: chairman (automatic), H. H. Kellogg, Columbia University; chairman-elect, A. E. Lee, Jr., American Metal Co. Ltd.; secretary, Robert W. Shearman, AIME; executive committee (1958 to 1961), H. W. St. Clair, USBM, and J. N. Anderson, Noranda Mines Ltd.

In the Institute of Metals Division the following have been nominated and will serve in 1958: chairman, W. J. Harris, Jr., Battelle Memorial Institute; senior vice chairman, O. T. Marzke, Naval Research Laboratory; vice chairman, T. A. Read, University of Illinois; and secretary, R. W. Shearman, AIME. Those on the executive committee to serve for three years are: C. E. Birchenall, James Forrestal Research Center, Princeton University; G. A. Roberts, Vanadium Alloy Steel Co.; and David Swan, Metals Research Laboratories, Electro Metallurgical Co.

The ISD Nominating Committee has submitted the following list of candidates: chairman, Karl L. Fetters; vice chairmen for one year each, L. R. Berner, H. M. Kraner, and M. Tenenbaum; executive committee to serve three years each, D. L. Clark, J. F. Elliott, and B. R. Queneau.

AIME Colorado Section Plans Rocky Mountain Minerals Conference

Sponsored by the AIME Colorado Section, the Rocky Mountain Minerals Conference will be held in Denver Oct. 30, 31, and Nov. 1, 1957. Tentatively scheduled is a symposium on uranium metallurgy which

(Continued on page 1000)

The Metallurgical Society of AIME Announces Nominations for 1958 Officers

The Nominating Committee of The Metallurgical Society of AIME has announced the nomination of John Chipman as Vice President of the Society for 1958. The Vice President automatically becomes President of the Society in 1959.

The Extractive Metallurgy Division has nominated the following men to serve on The Metallurgical Society Board of Directors: H. W. St. Clair for one year, C. C. Long to serve two years, and T. D. Jones for a three-year term.

The Institute of Metals Division nominees to the Board are: C. S. Smith, one year; J. B. Austin, two years; and J. S. Smart, Jr., three years.

In the Iron and Steel Division nominees will each serve on The Metallurgical Society Board for one year. They are: K. L. Fetters, H. B. Emerick, and K. C. McCutcheon.

Nominations to The Metallurgical Society Nominating Committee from EMD are J. C. Kinnear, Jr., C. C. Long, and T. D. Jones. IMD has nominated R. S. Busk, O. T. Marzke, and Robert Maddin to serve on the Nominating Committee. Those who have been chosen to represent ISD on the Nominating Committee are: H. B. Emerick, two years; V. W. Jones, one year; and T. S. Washburn, who will be serving the second year of a two-year term.

The candidate for Vice President, John Chipman, is Professor of Metallurgy at Massachusetts Institute of Technology where he has been teaching for 20 years. A native of Florida, Dr. Chipman was born Apr. 25, 1897, in Tallahassee. He holds an M.S. degree from the State University of Iowa, a Ph.D. from the University of California in 1926, and a Sc. D. from the University of the South. His teaching career began at the Georgia School of Technology. Dr. Chipman was associate director of research, American Rolling Mill Co., for three years before joining the staff of MIT in 1937. He became

JOHN CHIPMAN

head of MIT's Dept. of Metallurgy in 1946. Dr. Chipman has received numerous awards, among them the AIME Hunt Award, the ASM Howe Medal and Sauveur Achievement Award, the Clamer Medal of the Franklin Inst., and recognition from Italian and Swedish metallurgical societies. He was the AIME Howe Memorial lecturer in 1949.

New Names for ISD And IMD Committees

The Iron and Steel Division of the Metallurgical Society of AIME announces a change of name for one of its committees. In the future the Converter Steel Committee will be known as the Acid Converter and Basic Oxygen Steel Committee.

In the Institute of Metals Division the Metal Physics Committee has changed its name to Chemistry and Physics of Metals Committee.

Nominations Are Announced for 1958 AIME Officers

Three Society Nominating Committees Present Slate for 1958 Institute Officers and Directors

For President-Elect

H. C. PYLE

Howard C. Pyle has been nominated to serve as AIME President-Elect in 1958. Mr. Pyle has been nominated by the Society of Petroleum Engineers of AIME, the Society designated to nominate the 1959 President of AIME. The Society of Mining Engineers will designate the 1960 President, and The Metallurgical Society the 1961 President.

Born in Williams, Ariz., Mr. Pyle graduated from the University of California with a B.S. degree in petroleum engineering and geology in 1926, later obtaining professional and masters degrees in petroleum engineering from the University of Southern California. Chairman of the AIME Petroleum Branch in 1947, Mr. Pyle has been serving the Institute as a director since 1955. President, director, and chairman of the executive committee, Monterey Oil Co., since 1952, he is also director of the American Petroleum Inst. and the Western Oil & Gas Assn. During World War II he served as Captain in the office of Chief Engineers, and was sent to England on assignment to the General Staff of Supreme Commander Eisenhower. In the early days of the invasion, he was in Normandy to coordinate the initial imports of bulk fuel. Promoted to Lieutenant Colonel in 1944, he returned home after V-E Day. From 1945 to 1947, as vice president, Bank of America, Mr. Pyle was in charge of the bank's oil industry loans. He served as president of Continental Consolidated Corp. from 1947 to 1950 before opening his own office as consulting engineer in Los Angeles. From 1951 to 1952 Mr. Pyle was president and director of Jergins Oil Co. A member of the Conservation Committee of California Oil Producers Committee, and its Allocation Committee, he served this organization as chairman in 1942. Mr. Pyle has had many of his technical papers published by other technical societies as well as by AIME.

For Vice Presidents and Directors

Arthur W. Thornton, assistant to the vice president-operations, National Tube Div., U. S. Steel Corp., Pittsburgh, has been nominated by The Metallurgical Society of AIME as AIME Vice President for one year. The Society of Mining Engineers and the Society of Petroleum Engineers are each represented by two incumbent Vice Presidents. Currently a Past-President of The Metallurgical Society of AIME and Director of AIME, he also served as chairman of the Metals Branch Council during 1956-1957, taking an active part in the organization of the new Society. His efforts were directed toward developing and correcting The Metallurgical Society Bylaws; detecting their structural flaws, he aided in the solution of drafting problems. Mr. Thornton is a former chairman of ISD and has served on the executive committee of the NOHC and on the AIME Bessemer Committee (also the AIME Converter Steel Committee, and now the Acid Converter and Basic Oxygen Steel Committee). A native of McKeesport, Pa., Mr. Thornton graduated from Lehigh University in 1931 with a degree in metallurgical engineering. He was associated with the Weirton Steel Co., Weirton, W. Va., before joining the metallurgical department of Duquesne Works, U. S. Steel Corp. Since 1934 he has been with the National Tube Div., assuming his present post in 1954.

A. W. THORNTON

J. C. KINNEAR, JR.

John C. Kinnear, Jr., general manager, Nevada Mines Div., Kennecott Copper Corp., McGill, has been nominated by The Metallurgical Society to serve as AIME Vice President for one year and as a Director for three years. He is the first President of The Metallurgical Society. A native of Nevada, Mr. Kinnear was born in McGill in 1914 and graduated from Pomona College, Claremont, Calif., with a B.A. degree. He went on to obtain a B.S. in metallurgy in 1938 at Massachusetts Institute of Technology, where he first joined AIME as a Student Associate. During his summer vacations he worked for Nevada Consolidated Copper Corp. Mr. Kinnear's main professional association has been with Kennecott Copper, beginning in the company's McGill Smelter.

Transferring to the Chino Mines Div., Hurley, N. M., in 1939, Mr. Kinnear was present at the blowing in of the Hurley smelter. He became assistant superintendent of the smelter in 1942. Promoted to assistant to the general manager, Mr. Kinnear returned to McGill, Nev., in 1948 and became assistant general manager, Nevada Mines Div., in 1949. He assumed his present duties as general manager in 1950. Active in AIME both on the Local Section and National levels, Mr. Kinnear has served as chairman of the Nevada Section and as chairman of EMD.

For Directors

Elmer A. Jones, first President of the Society of Mining Engineers of AIME, has been nominated by that

The chairmen of the three Society Nominating Committees have announced nominations for AIME President-Elect, Vice Presidents, and Directors for 1958.

Each of the Society Bylaws provides for additional nominations. (The Bylaws of each Society in substantially final form have been published in the Journals and in revised form in the soon to be published 1957 Institute Directory.) If no alternate slate is proposed, no letter ballot will be necessary and the selections of the three Nominating Committees will be declared elected in accordance with the respective Society Bylaws well in advance of their installation at the 1958 Annual Meeting in February.

Society to serve as AIME Director for three years. Manager of Southeast Missouri Div., St. Joseph Lead Co., Mr. Jones is a native of Minneapolis where he was born in 1902. After graduating from the University of Minnesota School of Mines in 1924 with a degree in mining engineering, he began his career with the Minnesota State Highway. From 1925-1926 he worked for what is now part of Tennessee Copper Co. in Ducktown, Tenn.

Elmer Jones began his long association with St. Joe in 1926. Before assuming his present position he had served the company successively as mine surveyor, safety inspector, mine engineer, and assistant general mine superintendent. Two of his AIME technical publications have dealt with the subject of mine mechanization at St. Joe.

The second Director representing the Society of Mining Engineers will be the 1958 President of the Society. At press time for JOURNAL OF METALS, the future president of the Society of Mining Engineers of AIME had not been named.

E. A. JONES

B. P. KANTZER

Basil P. Kantzer, vice president in charge of the Gulf Div., Union Oil Co. of California, has been nominated by the Society of Petroleum Engineers of AIME to serve as AIME Director for three years. Mr. Kantzer will become President of the Society of Petroleum Engineers in 1958. Born in Salt Lake City, he received his B.S. degree in petroleum engineering from Stanford University in 1934. Mr. Kantzer began his engineering career with Union Oil. Progressing rapidly, he was promoted to division foreman at Bakersfield, Calif., in 1942, and chief production engineer in 1943, becoming manager of the California field operations in 1948. Mr. Kantzer assumed the post of manager of natural gas and gasoline for the entire company in 1950. By 1955 he was managing operations for Union Oil's Gulf Div. with headquarters in Houston. He has served as Pacific Coast Local Section Chairman for AIME.

John P. Hammond, assistant general superintendent, Production Dept., Amerada Petroleum Corp., has been nominated by the Society of Petro-

J. P. HAMMOND

W. R. HIBBARD, JR.

leum Engineers of AIME as AIME Director for three years. He is now President of the Society. A native of Oklahoma, Mr. Hammond graduated from the University of Tulsa in 1932, at which time he joined his father as an independent oil operator. However, in 1941 he took the opportunity to join Amerada, working for the company in Louisiana and New Mexico. In 1943 he was transferred to the Tulsa headquarters as assistant to the chief engineer, serving in this capacity until 1949, when he became assistant to the vice president in charge of production. Mr. Hammond assumed his present post in 1951. He has led an active professional life, serving AIME as chairman, Mid-Continent Section, Petroleum Branch Publications Committee, and was vice chairman, Petroleum Branch, in 1953.

Walter R. Hibbard, Jr., manager of alloy studies, General Electric Research Laboratory, Schenectady, has been nominated by The Metallurgical Society as AIME Director for three years. He is now serving as vice president of The Metallurgical Society of AIME, and chairman of IMD for 1957 and will be President of The Metallurgical Society in 1958. A native of Bridgeport, Conn., Dr. Hibbard graduated from Wesleyan University in 1939 with a degree in physical chemistry, receiving his doctorate in physical metallurgy from Yale University in 1942. After three years with the metallurgical section of the Bureau of Ships, USN, he returned to Yale as an assistant professor. He is presently an adjunct professor of metallurgical engineering at Rensselaer Polytechnic Institute Dr. Hibbard joined the staff of General Electric's Research Laboratory in 1951. He received the AIME Rossiter W. Raymond Award in 1950.

Incumbent AIME Officers and Directors as of March 1958:

The President of AIME for 1958 will be Augustus B. Kinzel.

Past-President in 1958 will be Grover J. Holt.

Vice Presidents for 1958 will be R. V. Pierce and J. L. Gillson, *Society of Mining Engineers;* and E. C. Babson and L. E. Elkins, *Society of Petroleum Engineers.*

The following will continue to serve as AIME Directors for 1958: A. Fletcher, F. J. Meek, L. Campbell, F. Strandberg, and Lamar Weaver, *Society of Mining Engineers;* C. R. Dodson, G. F. Moulton, and T. C. Frick, *Society of Petroleum Engineers;* R. B. Caples, C. R. Kuzell, A. W. Thornton, and J. S. Vanick, *The Metallurgical Society.*

AIME Mining Engineers Welcome New Secretary

Effective July 1, John Cameron Fox became Secretary of the Society of Mining Engineers of AIME, taking the position vacated by Arnold Buzzalini who resigned to return to private industry.

A member of the Institute since 1938, the incoming Secretary of the Society of Mining Engineers is well acquainted with members of the profession in every branch of the industry, and brings to his new job a broad understanding of industry operating and economic problems.

Since graduating from Columbia University School of Mines, Mr. Fox

has held a variety of jobs in the mineral industry which have taken him to all parts of the U. S., as well as to Latin America and Canada. He has been connected with mining operations in gold, lead, zinc, copper, nickel, and coal. Mr. Fox taught at his alma mater and also worked for several mining publications, serving as editor of *Mining Congress Journal* for five years.

French Metallurgical Society Plans Meeting

The French Metallurgical Society will hold its annual fall meeting in Paris from Oct. 7 to 12, 1957. The preliminary program containing resumes of the papers to be presented will be distributed in July.

Those interested in attending should contact M. J. Pomey, President, Societe Francaise de Metallurgie, 25 rue de Clichy, Paris 9e, France.

133 Attend Annual New England Regional Conference Held in Boston May 17 and 18

The Eleventh Annual AIME New England Regional Conference was held at the Hotel Statler, Boston, on May 17-18, 1957. The unusually fine technical program, with review papers on metal science and on metal engineering, was attended by 133.

Chairman W. A. Backofen, of Massachusetts Institute of Technology, opened the Conference on Friday morning. Chairman of the Metal Science Session on *Melting and Solidification* was P. J. Clemm, Knolls Research Laboratory, General Electric Co. K. A. Jackson of Harvard University presented a theoretical paper on *Solidification of Alloys*. This was followed by a paper on *Gases in Metals* by C. M. Adams of MIT. The third paper of the morning session, by J. Walker of General Electric Co., was on *Principles of Grain Refinement in Castings*. A pleasant interlude was the midway intermission for coffee.

The main event for Friday afternoon was a plant visit to the Measurements Laboratory, Instruments Dept., General Electric Co., Lynn, Mass. The group was welcomed by Thomas Paine, manager of the Instruments Dept. Ray Lever, manager, Materials & Methods Laboratory, briefed the visitors on their plant tour by presenting a talk and slides on metallurgical and ceramic problems. Small parties with guides then toured the laboratory areas devoted to applied physics, magnetics, reactor instruments, and primary standards. In the adjoining plant, the visitors were shown the manufacture of tiny jewels for instrument bearings, and photocells for exposure meters.

After returning to Boston, the Fellowship Dinner was held in the Bay State Room of the Hotel Statler, with A. Dudley Bach, president of New England Metallurgical Corp., serving as toastmaster. Brief remarks were given by Walter R. Hibbard, Jr., chairman of the Institute of Metals Division, and Vice President of The Metallurgical Society of AIME, and by R. W. Shearman, Secretary of the Metallurgical Society. Speaker of the evening was the Rev. Daniel Linehan, S.J., Western College Seismological Observatory, whose topic was *Operation Deep Freeze*. Father Linehan had accompanied the three expeditions made to the Antarctic in 1954, 1955, and 1956, in connection with America's contributions to the International Geophysical Year. Father Linehan served both as a geophysicist and as chaplain. The first trip in 1954 was made in connection with the scouting expedition of the icebreaker *Atka*. With the aid of excellent colored slides, Father Linehan told of the geography, geology, meteorology, fauna, and the problems of human life on Antarctica, affording the audience a real appreciation of the stark beauty of the area and the difficulties encountered by other explorers.

Following the formal dinner, an informal session on personal financial management was conducted. A motion picture on investments produced by the New York Stock Exchange, was shown, after which Harry Besse, president of the Boston Stock Exchange, spoke on *Investments, Good and Bad, for the Average Man*, and answered a host of questions from the audience on financial matters.

The Metal Engineering Session was held on Saturday morning, May 18, with R. M. Treco, Olin Mathieson Co., presiding. The first paper by W. W. Drykacz of Allegheny Ludlum Steel Corp. on *Production Vacuum Melting* summarized three methods of vacuum melting. His talk concluded with a film showing high-speed motion pictures of the consumable electrode melting process taken at 4000 frames per second.

Rufus Easton of Koppers Co. Inc., presented the second paper, *Recent Developments in Continuous Casting*. The speaker described the various processes that have been developed or proposed for continuous casting of various metals.

The last speaker, B. H. Alexander of CBS-Hytron, presented an excellent review of the *Metallurgy of Transistors*. An interesting parallel was drawn between the zone melting techniques used for producing elements for transistors in small quantities but at high purities, described by Dr. Alexander, and the consumable electrode process described earlier by Mr. Drykacz for producing larger quantities of very pure metals and alloys.

A business meeting of the New England Conference Committee was held on Saturday afternoon. Officers elected to serve for the Twelfth Annual Conference in 1958 were: Delmar E. Trout, chairman; W. A. Backofen, past-chairman; D. L. Martin, vice chairman; J. J. Burke, secretary; and J. P. Lynch, treasurer. The proposed Conference site for next year is Schenectady. Section representatives for 1958 include Robert Marsh and K. T. Aust from eastern New York; F. S. Chudy and W. R. Johnson from Connecticut; and R. M. Treco, P. J. Clemm, and L. R. Frazier as members-at-large.

Contributing in large measure to the success of the 1957 Conference were W. A. Backofen, chairman; D. E. Trout, vice chairman; D. L. Martin, secretary; and Sidney Siegel, treasurer.

Rocky Mountain

(*Continued from page 997*)

should serve as a forum for reviewing the alternative methods now available for the extraction of uranium.

For this fourth annual meeting, E. H. Crabtree will be general chairman, assisted by R. L. Scott. Other conference committee chairmen are: L. J. Parkinson, program; C. L. Barker, arrangements; G. M. Wilfley, entertainment; Arthur L. Hill, finance; and Fred L. Smith, secretary. Mrs. J. Paul Harrison is chairman of the ladies' activities.

In the Colorado Section, Asher H. Patten is chairman; Truman H. Kuhn, vice chairman; and William L. Miles, Jr., secretary-treasurer.

EJC-ECPD Schedule Engineers Assembly

An Engineers General Assembly is scheduled to be held Thursday and Friday, Oct. 24 and 25, at the Statler Hotel, New York. It will be the first step toward the coordination of the activities of Engineers Joint Council and Engineers Council for Professional Development. The EJC-ECPD joint program committee has arranged a series of panel discussions on topics of mutual interest. The program will examine the following subjects: military service as a factor in professional development, the community college in technological education, the place of the engineer in industrial management, and new dimensions in postgraduate education for engineers.

Concluding the conference will be an assembly dinner celebrating the 25th anniversary of ECPD.

New Institute Offers Translations of Russian Technological Research

Pergamon Institute, a nonprofit-making foundation has recently been formed in New York. Its purpose is to make available to English-speaking scientists, doctors, and engineers from all UN countries the results of scientific, technological, and medical research and development in the Soviet Union and other countries in the Soviet orbit.

To be established is a translation panel of competent translators from Russian and other Slavic languages into English. The Institute will charge customers for services on a cooperative cost-sharing plan. It also hopes to encourage the teaching of the Russian languages, to sponsor and foster research into the organization of scientific information, and to publish monographs in English reviewing Soviet scientific activity.

AIME

BOARD OF DIRECTORS

Recent actions taken by the Institute Board of Directors.

► The Engineering Societies Personnel Service was established by the Four Founder Societies, and at one time part of the cost was underwritten by these societies. Currently the ESPS is a nonprofit but self-supporting corporation. A 1956 deficit was paid from a modest surplus and it is hoped that there will be no deficit for 1957. The current employment situation is entirely different from that when the Service was established. More than half of the employers are willing to pay the employment fee, whereas this had formerly been borne by the applicant. It is planned to change the contract entered into by a member with the ESPS in order to take into consideration that in most cases the employer will pay the fee. This change plus publicizing the service will be an added service to a larger number of AIME members. It was also suggested that the ESPS make its services available at some of the larger Institute meetings. These suggestions were approved.

► John C. Calhoun, Jr., chairman of the Council of Education of AIME, was appointed to represent the Institute at the Centennial Convocation of the National Education Assn., to be held in Philadelphia, July 3, 1957.

► It was voted that the members of the Rossiter W. Raymond Award Committee serve as the AIME Noble Prize Committee, with the chairman representing AIME on the intersociety Alfred Noble Joint Prize Committee.

► Syracuse University's request for establishment of a student chapter, and the proposed bylaws, were approved, with one revision in Article 2, Section IV of the bylaws.

Steel Committee Visits Byers Plant in Ambridge

Among those attending the meeting were, first row, left to right: L. A. Xidis, D. E. Krock, and E. B. Hughes, Wheeling Steel Corp., Steubenville, Ohio; A. J. Sottile, Bethlehem Steel Co., Sparrows Point; George McMorran, U. S. Steel Corp., Gary; A. C. Coulson, Wheeling Steel Corp., Steubenville, Ohio; J. M. Stone, U. S. Steel Corp., Fairfield, Ala; A. C. Flanders, Wheeling Steel Corp., Benwood, W. Va.; and George Harry, Jones & Laughlin Steel Corp., Aliquippa, Pa.

Second row, left to right: A. W. Thornton, National Tube Div., U. S. Steel Corp., Pittsburgh; A. J. Macdonald, Weirton Steel Co., Weirton, W. Va.; F. J. McMulkin, Dominion Foundries & Steel Co. Ltd., Hamilton, Ont., Canada; R. C. Dierker, National Tube Div., U. S. Steel Corp., Lorain, Ohio; M. G. Morrison, Youngstown Sheet & Tube Co., Youngstown; H. L. McFeaters, Pennsylvania Engineering Corp., New Castle, Pa.; W. H. Mathesius, Koppers Co. Inc., Pittsburgh; J. P. Ely, National Tube Div., U. S. Steel Corp., McKeesport, Pa.; G. M. Yocom, Wheeling Steel Corp., Steubenville, Ohio; and C. G. Mason, Republic Steel Corp., Youngstown.

Third row, left to right: J. F. O'Brien, U. S. Steel Corp., Chicago; J. A. Kirk, Bethlehem Steel Co., Sparrows Point; J. W. Zubaly, Jones & Laughlin Steel Corp., Aliquippa, Pa.; H. C. Dunkle, Republic Steel Corp., Youngstown; R. A. Beals, JOURNAL OF METALS, AIME, New York; L. R. Cederstrom, National Tube Div., U. S. Steel Corp., McKeesport, Pa.; J. I. Hopkins, Bethlehem Steel Co., Sparrows Point; R. W. Shearman, AIME, New York; Barney Dagan, Kaiser Steel Corp., Fontana, Calif.; L. J. Rohl, U. S. Steel Corp., Pittsburgh; and J. S. Gamble, National Tube Div., U. S. Steel Corp., McKeesport, Pa.

Fourth row, left to right: A. B. Wilder, National Tube Div., U. S. Steel Corp., Pittsburgh; E. P. Best, A. M. Byers Co., Ambridge, Pa.; S. J. Dougherty, Weirton Steel Co., Weirton, W. Va.; and Josef Reichmayr, Pennsylvania Engineering Corp., New Castle, Pa.

After assembling at the Penn-Sheraton Hotel, Pittsburgh, on the morning of May 15, the members of the AIME Converter Steel Committee went out to the Ambridge Plant of the A. M. Byers Co. The chairman of the committee this year, A. B. Wilder, headed the visit, and among the guests were A. W. Thornton, Past-President of The Metallurgical Society of AIME, and R. W. Shearman, Secretary.

The morning was devoted to touring the cupola melting, desulfurization, and converter operations. The guests also observed the production of wrought iron by the company's unique process. Following lunch at the Ambridge Country Club, the committee held an afternoon off-the-record technical meeting.

On arrival at the plant the guests heard a few words from their host, B. M. Byers, president, who introduced R. Frame, vice president of operations, and E. P. Best, chief metallurgist.

In the course of the afternoon meeting, the committee voted to revise its name to reflect the increasing variety of pneumatic steelmaking processes. The new name is AIME Acid Converter and Basic Oxygen Steel Committee; see p. 997.

New Thermophysical Properties Research Center at Purdue

Purdue University has announced the establishment of a Thermophysical Properties Research Center with two long-range objectives: 1) to serve as a world center for research and for the collection, analysis, correlation, and dissemination of data on thermophysical properties; and 2) to provide unique facilities and opportunities for graduate study and research in the field.

In this project, government agencies and industry are cooperating by providing multiple financial support. Each sponsor contributes approximately the amount of aid he might give an individual graduate student. At the same time, he is participating in and benefiting from a major research program covering a broad field of application.

The Thermophysical Properties Research Center was established after a careful two-year study of government, industry, and university needs for scientific information in the field.

The last phase of this study consisted of personal discussions with representatives of several nonprofit research foundations, numerous governmental research agencies, a score of industrial organizations, and the committees on thermophysical properties of two national technical societies.

The program is under the immediate direction of Y. S. Touloukian, professor of mechanical engineering. An advisory committee has been established to serve as a consulting body of experts and to act as a coordinating group for all research on thermophysical properties conducted at Purdue. Currently, Dr. Touloukian is forming a research staff consisting of trained and experienced personnel in physical chemistry, physics, metallurgy, chemical and mechanical engineering, and library science.

Annual Meeting of CIM in Ottawa Draws Large Attendance

Following the trend of the times in institute meetings, the 59th Annual General Meeting of the Canadian Institute of Mining and Metallurgy, held at the Chateau Laurier in Ottawa from April 22 to 24, was the largest annual meeting yet held. Attendance set a new record with 1307 men and 531 ladies present.

Representing AIME were President Grover J. Holt and Secretary Ernest Kirkendall, President Holt bringing greetings to the CIM at the Monday welcoming luncheon.

The technical sessions held during the meeting were numerous and well attended, with outstanding papers offered. Monday morning was devoted to divisional business conferences, and that afternoon a general session treated the subject of the St. Lawrence Seaway to which a field trip was scheduled the next day. In addition, a particularly informative color film of the project was shown.

St. Lawrence Seaway

The history of the effort to make the St. Lawrence an economic avenue of transportation goes back to 1700 when the first attempts were made to get a canal built at Lachine. From 1800 to 1848 a 9-ft canal system was completed. By 1901 a 14-ft system had replaced the earlier canals; and by 1959 the 27-ft draft system, the St. Lawrence Seaway, should be completed, after five years of construction. The principal commodities carried will be iron ore westward and grain eastward, but a through water route from the Atlantic to Great Lakes ports will offer more economic transportation for much other freight as well. No dearth of markets for the 2,200,000 hp that will be developed is now anticipated.

Tuesday morning simultaneous technical sessions were held on geology, industrial minerals, mineral dressing, iron and steel metallurgy, and ore and coal mining mechanization. In the afternoon there were also five sessions on coal, geology, petroleum, and natural gas, extractive metallurgy, fatigue of metals, and pillar mining. Wednesday morning there were again five separate sessions, this time on geology, industrial minerals, mineral dressing, metal mining, and mineralogy; and that afternoon the technical program featured two geological sessions.

A full social program had been arranged: a reception on Sunday night; pre-luncheon cocktails on Monday, Tuesday, and Wednesday for all registrants; a variety show Monday night, also attended by the distaff, which was followed by dancing; the annual dance and president's reception on Wednesday; and general luncheons all three days. In addition the ladies took a tour of the Parliament buildings, a drive through Ottawa, and also enjoyed two afternoon tea parties.

AIME Winner

Francis R. Joubin, an AIME member of Toronto, received the Selwyn G. Blaylock Medal for outstanding work in Canadian exploration geology during the last five years, and particularly for his "direction of the discovery and early development of the Blind River uranium field."

Chatting between sessions are, left to right: J. L. Ramsell, Kerr Addison Gold Mines Ltd.; Alan Scott, M. J. O'Brien Ltd.; J. M. Cunningham-Dunlop, Nipissing Mines Co. Ltd.; A. E. Cave, New Calumet Mines Ltd.; and AIME President Grover J. Holt.

New Petroleum Book

The price of *Statistics of Oil and Gas Development*, Vol. II, has been raised from $5.00 to $7.50 for members and from $10.00 to $15.00 for nonmembers. The old prices were listed in the December 1956 issue of JOURNAL OF METALS, in accordance with the old Bylaws of AIME which stipulated that prices of AIME publications appear in the December issue each year. However, at the meeting of the Executive Committee of the Petroleum Branch on Feb. 25, 1957, this price increase was voted. Approval of this increase was later voted by the AIME Executive Committee on Apr. 15, 1957.

Magnetism Conference Set for Nov. in Capitol

A Conference on Magnetism and Magnetic Materials is scheduled to be held Nov. 18 to 20, 1957, at the Hotel Sheraton-Park, Washington, D.C. The conference is sponsored by the AIEE, in cooperation with AIME, the American Physical Society, the Institute of Radio Engineers, and the Office of Naval Research.

Programs containing abstracts of all papers will be distributed before the meeting. Emphasis will be on the basic exploration of magnetic phenomena which have some bearing on potential engineering applications. The technical sessions will also concentrate on the development of apparatus and techniques which make use of recent advances in magnetism.

Further information may be obtained by writing the local conference committee chairman: L. R. Maxwell, U. S. Naval Ordnance Laboratory, Silver Spring, Md.

Prospective exhibitors should contact Davis S. Bender, 930 H St., N.W., Washington 1, D. C.

UET Grants Used for Engineering Research

Engineering research will go forward on a wide front with new grants made by Engineering Foundation, a department of United Engineering Trustees Inc., at its annual meeting held in New York on May 16. Appropriations for the 1957-1958 fiscal year total $69,000. They will initiate or advance 28 projects, which will receive nearly $1,000,000 in industry support. The projects to which funds have been allocated represent all the important branches of the profession. They are being carried out in university, government, and industrial laboratories all over the country under sponsorship of the major engineering societies.

Alloys of Iron Research is receiving $5000 to continue its work of

(Continued on page 1005)

AROUND THE SECTIONS

AIME President Grover J. Holt became an "Arkansas Traveler," an honor conferred upon distinguished visitors to the state. Shown left to right: Governor Faubus presenting the official certificate to Mr. Holt as AIME Field Secretary Roy O'Brien and Luther Branting, Alcoa, look on.

A highly successful spring tour covering many states and Canada was recently taken by AIME President Grover J. Holt, who visited Local Sections throughout the country, accompanied by AIME Secretary E. O. Kirkendall and Field Secretary Roy E. O'Brien.

First stop was Little Rock, Ark., on March 27, where Governor O. Faubus presented President Holt with an honorary certificate naming him an official "Arkansas Traveler." This visit to the Arkansas Section included tours of the Big Rock Stone & Material Co., Reynolds Mining Corp., and the Mining Div., Aluminum Co. of America.

The following day President Holt was guest of honor at the Tri-State Section Banquet in Miami, Okla. Following a reception in the mezzanine of Hotel Miami, President Holt addressed the dinner guests on the history of the iron ore industry from its infancy to the record breaking years of 1955-1956. He also urged encouragement of engineering students and a closer alliance between the mining industry and student chapters of engineers.

In El Paso, Texas, the Local Section held a dinner in his honor at the Hotel Cortez on March 29. President Holt spent the next two days in New Mexico, dividing his time between Silver City and Carlsbad, before leaving for Los Angeles to spend April 3 there. He arrived in Reno on April 5, in time for the Pacific Southwest Mineral Industry Conference, sponsored by the Nevada, San Francisco, and Southern California Sections, attending the cocktail party and dinner at the Riverside Hotel.

By April 8, he was back east in Pittsburgh, his presence honoring the National Open Hearth Steel and Blast Furnace, Coke Oven, and Raw Materials Conferences. Turning westward again, he flew to Portland, Ore., to attend the Pacific Northwest Regional Conference in the Multnomah Hotel on April 11. Then, heading north to visit our Canadian neighbors, President Holt represented AIME at CIM's annual meeting in Ottawa, April 22.

During the first two weeks in May, Mr. Holt's itinerary included stops at five local sections. On May 4, he was in the Windy City as the Chicago Section turned out in full force to welcome him. The Montana Section held a joint meeting with the Anderson-Carlisle Society in the Museum Hall, Montana School of Mines, on May 11. As featured speaker of the evening, President Holt discussed the iron ore industry, and also gave a brief report on the affairs of the Institute. On Monday, May 13, Mr. Holt spoke to the student body at the Montana School of Mines on the topic of research.

Coeur d'Alene Subsection of the Columbia Section was the next port of call for the roving AIME President. The meeting in his honor was held in Kellogg, Idaho, on May 14. One of the highlights during the welcoming dinner for Mr. Holt was the award to Arthur W. Fahrenwald of the honor "Inland Empire Engineer of the Year" for his outstanding achievements in the field of ore dressing. The next two days were spent in Washington where Mr. Holt made two stops, Spokane and Seattle, before winding up his tour in San Francisco on May 20. There, he attended a special President's Meeting in the Engineer's Club, and addressed the Section on the "new look" for AIME.

From San Francisco Mr. Holt planned to travel to Billings, Mont., where he would have attended another AIME meeting on his way east. He deplaned to stay overnight in Seattle, and coming into the city the

(Continued on page 1005)

President Grover J. Holt, right, was special guest at the luncheon meeting of the Chicago Section executive committee on May 4. Section Chairman Michael Tenenbaum, left, led the discussion on plans for Chicago's role as AIME host Section during the World Metallurgical Congress, Nov. 2-8, 1957. Another topic considered was the IMD meeting Nov. 4-7, and the ISD special activity meeting on Nov. 5, both slated to be held in the Windy City.

Personnel

(Continued from page 942)

applied research. Salary, $7980 to $9380 a year. Location, Midwest. W5032.

Physical Metallurgist. Excellent opportunity available for B.S. or M.S. with approximately five years experience. Duties will include studies of equipment failure and research on corrosion resistance and quality control. Will also supervise and correlate operations of testing laboratory. Salary, open. Location, Ohio. W5028.

Metallurgist, B.S. in metallurgical or chemical engineering, for quality control metallurgical laboratory, testing and analysis, preparation of operation sheets and work orders; stress, rupture, hardness, and other special tests. Salary, $6500 to $9000 a year. Location, New York metropolitan area. W5024.

Project Engineer, mechanical or metallurgical degree, with working knowledge and three to five years experience in hot metal forging and extrusion and basic working nonhot services, to direct activities of several process engineers and be responsible for recommendation and development and execution of methods and products; design and layout of required equipment, and tooling, for leading eastern manufacturer of extrusion products. Salary, open. Location, western New York. W5003.

Foundry Engineer, B.S. in metallurgy or metallurgical engineering, with experience in high temperature alloys and thorough knowledge of sand, shell ceramics, and permanent molding. Will be responsible for developing foundry processes, preparing specifications, controlling quality standards, and recommending production methods for leading eastern manufacturer producing stainless steel iron castings for reciprocating aircraft engines. U. S. citizen. Location, western New York. W5002.

Metallurgist, with ferrous and nonferrous experience in metallography, physical testing, etc., for commercial testing laboratory. Salary, $6000 to $7000 a year. Location, New York. W4997.

Engineers. a) *Physical Metallurgist,* B.S., M.S., or Ph.D. in metallurgy, with experience in basic steel or nonferrous metals industry, for research in development of new titanium-base alloys, heat treatment evaluation, corrosion control studies, and manufacturing processes. Salary, $5,400 to $10,200 a year. b) *Physical Chemist or Physical Metallurgist,* to conduct corrosion research studies on titanium and titanium-base alloys. Studies will include protective coatings as well as effects of composition modification and/or surface treatments on corrosion characteristics of alloys. Background or interest in electrowinning or electrorefining will also be advantageous. Salary, $6,000 to $10,200 a year. Company will pay placement fee. Location, Pennsylvania. W4978.

Associate Professor or Professor, to head department of metallurgy. Will do some teaching as well as independent and sponsored research. Salary, open. Location, New York metropolitan area. W4741.

Sales Engineer, graduate metallurgist, preferably with some experience in the mineral processing industry. Must speak Spanish and be free for quite extensive travel. Salary, open. Company pays placement fee. Headquarters, Midwest. W4724.

Professorial Chair, metallurgy or physics of metals; no administrative assignments. Salary to start, $15,000 a year. Available September 1958. Location, eastern U. S. W4860.

Agglomeration Engineer, for steel plant, graduate mechanical or metallurgical engineer, with ten years experience in field of agglomeration, preferably iron ore. Will coordinate, design, and prepare flow sheets, equipment lists, and cost estimates

(Continued on page 1005)

IMMEDIATE OPENING
CRUCIBLE STEEL COMPANY OF AMERICA
Central Research Laboratory
Pittsburgh, Pennsylvania
MECHANICAL ENGINEER
Requires advance degree in mechanical engineering or metallurgy with experience in both technical disciplines. Should have a minimum of ten years' experience in development of metal working processes and equipment. Competence in handling engineers associated with the working of metals is desirable.
Send resume in complete confidence to
Mr. E. J. Martin
Crucible Steel Company of America
P. O. Box 88
Pittsburgh 30, Pennsylvania

DIRECTOR OF RESEARCH AND DEVELOPMENT

A research organisation serving the Anglo American Corporation of South Africa Limited group's mines in Northern Rhodesia, require the services of a highly qualified and experienced Director of Research and Development to direct and coordinate laboratory and pilot scale investigations in the mineral dressing, extraction, metallurgy and refining of copper, cobalt, lead, zinc and other base metals.

As the post is a responsible one, applicants should be graduates in chemistry, metallurgy, chemical engineering, or allied fields, with emphasis on inorganic chemistry or metallurgy, and should have at least fifteen years total pertinent experience. In addition, experience of applied industrial research administration will be a recommendation.

Applicants should be between 35 and 50 years of age. Salary will be paid commensurate with qualifications and experience.

There are generous pension and leave privileges, and excellent housing accommodation is provided at nominal cost in a modern community.

Selected candidates will be interviewed personally before an appointment is decided.

Applications, giving personal and professional particulars, enclosing photograph and references, should be addressed to the Personnel Officer (Salisbury), Anglo American Corporation of South Africa Limited, P.O. Box 1108, Salisbury, Southern Rhodesia.

RESEARCH METALLURGIST

Physical Metallurgist for basic research in the behavior of metals at elevated temperatures. Ph.D. with up to 5 years research experience.

Company sponsored research program with a liberal publication policy. Excellent experimental facilities. Good opportunity for professional advancement.

Write or Phone
W. A. JOHNSON
Associate Director
of Research
Thompson Products, Inc.
23555 Euclid Avenue
Cleveland 17, Ohio

IMMEDIATE OPENING
CRUCIBLE STEEL COMPANY OF AMERICA
Central Research Laboratory
Pittsburgh, Pennsylvania
CERAMIST
Requires advanced degree in ceramics or physical chemistry coupled with at least ten years' experience in refractories, powder metallurgy, magnetic materials, etc., to direct challenging research problems in these fields.
Send resume in complete confidence
Mr. E. J. Martin
Crucible Steel Company of America
P. O. Box 88
Pittsburgh 30, Pennsylvania

Personnel

(Continued on page 1005)

for agglomerating facilities. Salary, to $1000 a month. Location, San Francisco. S2894.

Metallurgical Engineer, preferably B.S. or M.S., with knowledge of selective flotation, leaching, and electrolysis, for research, ore testing, and process controls. Salary, open. Location, Idaho. S2845.

Assistant Professor or Instructor, to teach physical metallurgy or related courses in metallurgy department of large engineering school. Nine-month appointment; opportunity for graduate work for personnel with instructor's rank. Location, Midwest. W5084.

IMMEDIATE OPENING

CRUCIBLE STEEL COMPANY OF AMERICA

Central Research Laboratory
Pittsburgh, Pennsylvania

APPLIED RESEARCH PHYSICIST

Requires advanced degree in physics or mathematics to undertake high level research activity in solid state of ferrous alloy systems and nonmetallic magnetic materials.

Send resume in complete confidence

Mr. E. J. Martin
Crucible Steel Company of America
P. O. Box 88
Pittsburgh 30, Pennsylvania

TECHNICAL SERVICE REPRESENTATIVE

Engineer required by major national supplier. Interesting and diversified work with sales and production groups to increase acceptance of company products. Location New York City with travel away from headquarters averaging up to 40-50% of the time, usually trips of short duration. Prefer graduate engineer with sales ability. Opportunity to become associated on permanent basis with major corporation, oldest and largest in its field. Liberal benefits. Salary $5000-8500, depending on ability and experience. Submit complete resumé in confidence including salary requirements

to: Box 8-JM AIME

For additional Engineering Societies Personnel Service items, see p. 942. Classified advertising also appears on pp. 942, 943, 944, 945, 948, and 1004.

Around the Sections

(Continued from page 1003)

airport limousine in which he was riding was involved in a serious accident hospitalizing many of the passengers. Although his injuries forced him to cancel his attendance at the Billings meeting, his many friends will be glad to hear that he has completely recovered from the effects of the crash, except for a slight scar on his forehead.

• The **St. Louis Section** held a joint meeting with the Engineers Club of St. Louis on March 28, at which time Edward L. Clark spoke on *Uranium Hunting.* Vice president of exploration and development for Four Corners Uranium Corp., Grand Junction, Colo., Dr. Clark presented a brief geological review of the major uranium areas in the U. S.

The Section's Annual Dinner Dance was held on May 10, at the Missouri Athletic Club. Preceded by a cocktail party, the affair this year honored AIME Vice President Roger V. Pierce.

• At the **Cleveland Section** dinner-meeting on May 16 in the Manger Hotel, members and guests enjoyed a program arranged by the McDowell Co. After cocktails and dinner, H. E. Rowen, general manager of the company, addressed the group and introduced the featured speaker of the evening, T. E. Ban, who discussed agglomeration techniques including the Dwight-Lloyd sintering system. Mr. Ban, who is director of research at McDowell, also talked about research in progress. A sound color film, *Giants of Industry,* on the ore handling and processing machinery manufactured by Wellman, concluded the program.

• A large turnout of miners and geologists marked the second annual uranium symposium of the **Uranium Section** April 5 to 7 in Salt Lake City. Among the principal speakers were Alan Jones of the AEC in Grand Junction, Colo., who reviewed *Outlook For Uranium,* and G. T. Harley, consulting geologist, who discussed the economics of *Potash in the Paradox Basin.*

• The Institute of Metals Division of the **Pittsburgh Section** met on May 16 at Carnegie Institute of Technology where E. S. Davenport was guest speaker. Assistant to the vice president, U. S. Steel Corp., Mr. Davenport spoke on *Some Observations on the Ferrite-Carbide Aggregates in Alloy Steels.*

The Section was co-sponsor with the National Open Hearth Steel Committee of an annual golf party on June 10, at the South Hills Country Club, Allegheny County, Pa. In addition to playing golf, guests also had the opportunity to cool off in the swimming pool.

• The AIME, NOHC **Western Section** decided to hold a field trip instead of its regular May dinner-meeting. Members visited the U. S. Lime Products Corp. in the Las Vegas, Nev., area on May 10, and returned home on May 12.

• The **Adirondack Section** held its first meeting of the year at Republic Steel Corp., Lyon Mt., N. Y., on April 27. Members had a choice of an underground tour or a trip through the mill and sintering plant. In the evening dinner was served in the American Legion Home, at which time Allan Mogensen gave a talk on *Work Simplification Programs.* Newly elected Section officers for 1957 are: A. L. Hall, chairman; M. Lowry, vice chairman; and C. Dievendorf, secretary-treasurer.

• The **Niagara Frontier Section** held a joint meeting with the CIM and a Canadian field trip on April 3. Members visited Port Colborne Works of International Nickel Co. of Canada in the afternoon. Later they met for a fellowship hour at the Hotel Barclay in Welland, Ont., which was followed by dinner. Featured speaker of the evening was W. J. Cook, assistant works manager at Inco's Port Colborne Works. His topic was *Nickel Refining.*

UET Grants

(Continued from page 1002)

publishing monographs devoted to important findings on carbon and alloy steels and cast irons. This June the project issued the fourth monograph of a new series, entitled *Boron, Calcium, Columbium, Tantalum, and Zirconium in Iron and Steel,* which is of much interest to the manufacturers of jet engines and guided missiles. The new monograph contains a summary of valuable material on the complex high-temperature alloys that have been developed in the past five or ten years. It is these new alloys which are making jet engines and more efficient power plants possible because they can withstand higher temperatures than ordinary steel. Alloys of Iron Research has been receiving Engineering Foundation support since its formation in 1929. It is a project sponsored by AIME.

An allocation of $4000 goes to the Corrosion Research Council—also an AIME-sponsored project—which completed its first year on Dec. 31, 1956. One of the scientifically important facts already revealed by the Council's first program is that light slows down the rate of corrosion in water containing air. Furthermore, exposure to a beam of light apparently causes the oxide film on metal to dissolve. This research is being done in cooperation with the National Bureau of Standards.

PERSONALS

Arthur Tix, winner of the AIME Robert W. Hunt Silver Medal, was unable to attend the Annual Meeting in New Orleans. The award was presented to Dr. Tix on May 13 at the Bochumer Verein Society in Dusseldorf, Germany, by the American Consul General.

A. TIX

John Cattanach, Jr., has been appointed manager of sales for tubular products and cold finished steel bars, Joseph T. Ryerson & Son Inc., Buffalo. Prior to joining Ryerson in 1933, Mr. Cattanach was associated with Genesee Bridge Co., Rochester, N. Y.

John Price Hirth, a student at Carnegie Institute of Technology, has been awarded a Fulbright scholarship to study metallurgy at the University of Bristol, England.

Roman Smoluchowski, professor of physics and metallurgical engineering, Carnegie Institute of Technology, Pittsburgh, will give a series of lectures in July at the International Summer School on Solids in Varenna, Italy. For the past five years Dr. Smoluchowski has been engaged in preparing an AEC report on *Effects of Irradiation in Atomic Reactors.*

Wilfred H. Stewart has been appointed staff engineer at Sunbeam Corp.'s Industrial Furnace Div., Chicago. Prior to joining Sunbeam, Mr. Stewart was executive engineer with the U. S. Reduction Co. For the past 17 years, he has been engaged in furnace design, refractory research, and the melting and smelting of aluminum and other light metals.

A. A. Wallach, former chief metallurgist, San Manuel Copper Corp., San Manuel, Ariz., has joined Kaiser Engineers' Development Div., Oakland, Calif.

H. Gordon Poole, head of the Dept. of Metallurgical Engineering, Colorado School of Mines, has been appointed research and development consultant for Oregon Metallurgical Corp.'s operations in Albany, Ore. Formerly associated with the USBM, Dr. Poole is currently supervising research for the Naval Bureau of Ordnance.

Eugene G. Grace, chairman of Bethlehem Steel Co., has been awarded a citation by the trustees of Lehigh University in recognition of his 44 years' service as a university trustee. Mr. Grace, Charles F. Rand Memorial medalist for 1948, was named president of the Lehigh board of trustees in 1924, and served in that capacity until 1956, when the board elected him honorary chairman.

B. R. Nijhawan has been appointed director of the National Metallurgical Laboratory, Council of Scientific and Industrial Research, Jamshedpur, India. He was recently elected a fellow of the National Institute of Sciences of India.

John H. Baisley has become manager, metals industry sales, for the Pittsburgh office of Allis-Chalmers Industries Group. A graduate of the University of New Mexico, where he received degrees in electrical and mechanical engineering, Mr. Baisley joined Allis-Chalmers as sales representative in 1950.

Stewart G. Fletcher, formerly director of metallurgy, Latrobe Steel Co., Latrobe, Pa., has been promoted to vice president-metallurgy. A frequent lecturer in the field of tool and die steels, Dr. Fletcher has twice been awarded the Howe Medal by ASM for research in steel heat treatment.

Addison Davidson, Jr., was recently appointed eastern sales manager for Femco Inc., Quakertown, Pa. He has been associated with the company for eight years in the capacity of sales engineer.

Lewis E. Thelin has been promoted to manager of technical service, Bristol Brass Corp., Bristol, Conn. He was formerly assistant chief metallurgist for the company.

STEWART G. FLETCHER

W. A. DEAN

Walter A. Dean has been promoted to assistant development metallurgist at Aluminum Co. of America. Formerly chief metallurgist for Alcoa's Cleveland works, Dr. Dean will now be located at the company headquarters in Pittsburgh. He has been with Alcoa since 1929 when he joined the staff of the Research Laboratories at New Kensington, Pa. **Thomas R. Gauthier** succeeds Dr. Dean at Cleveland.

K. M. Bartlett was recently elected president of Horizons Inc., Cleveland. Prior to joining Horizons as executive vice president in 1955, Mr. Bartlett had been associated with Thompson Products Co., Cleveland. He has been a member of various NACA, Navy, and Materials Advisory Board committees, and was founder and first president of the Investment Casting Institute.

Two AIME members were recently honored by the Steel Founders' Society of America at its 55th annual meeting. **Henry D. Phillips,** executive vice president, Adirondack Foundries & Steel Inc., Watervliet, N. Y., and retiring chairman of the steel society's technical research committee, was awarded the Technical & Operating Gold Medal. This was in recognition of his services as chairman of the committee over a five-year period and for activities which have benefited this country and the industry. During World War II, Mr. Phillips directed the steel casting activities of the Bureau of Ships, USN, and was instrumental in the revision of many specifications made necessary by shortages in raw materials. **E. E. Hucke,** assistant professor of chemistry and metallurgical engineering, University of Michigan, received the Gustav A. Lilliequist Steel Foundry Facts First Place Award for his article *High Amperage Magnaflux Inspection.* Mr. Phillips addressed the Society on *Steel Castings and Technical Research.*

Louis F. Sattele has been transferred from the Lorain, Ohio, office, Na-

tional Tube Div., U. S. Steel Corp., to National Works, McKeesport, Pa., in the capacity of general superintendent.

F. E. Van Voris has become metallurgical service representative in the open hearth section, Electro Metallurgical Co., Cleveland. Prior to his appointment, Mr. Van Voris had been employed for 17 years by Bethlehem Steel Co., where he was engaged in steel research and open hearth operations.

John H. Bassarear has been appointed mill superintendent at National Lead Co.'s MacIntyre Development, Tahawus, N. Y. Prior to his appointment, Mr. Bassarear was sinter plant superintendent.

I. L. Barker has transferred to the New York office of Cerro de Pasco Corp. He was formerly located in La Oroya, Peru.

Edward E. Slowter has been elected a vice president of Battelle Memorial Institute, Columbus, Ohio. Prior to his new appointment, Mr. Slowter was secretary and business manager.

OBITUARIES

Howard R. Youngkrantz (Member 1935) died on March 27, at the age of 43. A native of Chicago, he studied chemical engineering at Illinois Institute of Technology, beginning his lifetime career with Apex Smelting Co. upon graduation in 1935. From assistant metallurgist he progressed to chief metallurgist, finally becoming chief engineer. His professional affiliations included the American Institute of Physics, the American Foundrymen's Society, and ASM.

Carl Hans Vom Baur (Member 1954) died on May 28 at his home in New York. A pioneer in the development of electric furnaces, Mr. Vom Baur was the author of a standard textbook *Electro-Furnaces in the Iron and Steel Industry*. Born in New York in 1878, he attended Columbia University, graduating in 1899 with a degree in electrical engineering. He also took special courses at Massachusetts Institute of Technology, Brooklyn Polytechnic Institute, and studied in Darmstadt, Germany. For ten years Mr. Vom Baur had been an electrical engineer for the Board of Water Supply, and prior to that had been consultant to the Army Arsenal, Watertown, Mass., the Naval Reserve, and to the governments of Russia, Mexico, and Portugal. He was the holder of patents for his improvements in electric furnaces. At one time he had been engaged in the manufacture of furnaces in addition to consulting work.

NECROLOGY

Date Elected	Name	Date of Death
1913	Waldo H. Comins	July 26, 1956
1951	William R. Dunn	Unknown
1956	George H. Evans	Sept. 30, 1956
1928	George W. Greenwood	March 1957
1943	Otto Haentjens	Mar. 31, 1957
1905	Cornelius F. Kelley Legion of Honor	May 12, 1957
1932	Frank H. Reed	Apr. 27, 1957
1954	Carl H. Vom Baur	May 28, 1957
1952	Frank Weir	Unknown
1913	Walter C. Mendenhall Honorary Member	June 2, 1957

MEMBERSHIP

Proposed for Membership

Metallurgical Society of AIME

Total AIME membership on May 31, 1957, was 27,379; in addition 2,972 Student Associates were enrolled.

ADMISSIONS COMMITTEE

Harold K. Work, Chairman; Francis B. Foley, T. D. Jones; E. S. Machlin; J. H. Scaff, Paul Von Stein, T. B. Winkler.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

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COMING EVENTS

Sept. 8-Oct. 9, Commonwealth Mining and Metallurgical Congress, British Columbia to Nova Scotia.

Sept. 13, AIME St. Louis Section, joint meeting with Amer. Chemical Soc. St. Louis Section, Hotel York, St. Louis.

Oct. 3-4, AIME, NOHC Southwestern Local Section, fall meeting, Shamrock Hotel, Houston.

Oct. 6-10, Electrochemical Soc., Statler Hotel, Buffalo.

Oct. 7-12, Societe Francaise de Metallurgie, annual fall meeting, Paris, France.

Oct. 10-11, Joint Solid Fuels Conference, ASME-AIME Coal Div., Chateau Frontenac, Quebec.

Oct. 11, AIME, NOHC Eastern Local Section, 11th annual technical meeting, Sheraton Hotel, Philadelphia.

Oct. 18-19, AIME, NOHC Southern Ohio Local Section, annual meeting, Deshler-Hilton Hotel, Columbus, Ohio.

Oct. 30-Nov. 1, AIME Rocky Mountain Minerals Conference, Denver.

Nov. 1, AIME Pittsburgh Section and NOHC Pittsburgh Section, 12th annual Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh.

Nov. 2-8, Second World Metallurgical Congress, Chicago.

Nov. 3-7, AIME, IMD Fall Meeting Morrison Hotel, Chicago.

Nov. 5, AIME, Iron and Steel Division, Technical Sessions, Morrison Hotel, Chicago.

Nov. 12, AIME, NOHC Buffalo Local Section, annual meeting, Royal Connaught Hotel, Hamilton, Ont., Canada.

Dec. 4-6, AIME, 15th Electric Furnace Steel Conference, Penn-Sheraton Hotel, Pittsburgh.

Feb. 16-20, 1958, AIME Annual Meeting, Statler and Sheraton-McAlpin Hotels, New York.

Apr. 14-16, AIME, 41st National Open Hearth Steel and Blast Furnace, Coke Oven, and Raw Materials Conference, Penn-Sheraton Hotel, Pittsburgh.

May 5-6, AIME, Conference on Properties of High-Strength Steel, Penn-Sheraton Hotel, Pittsburgh.

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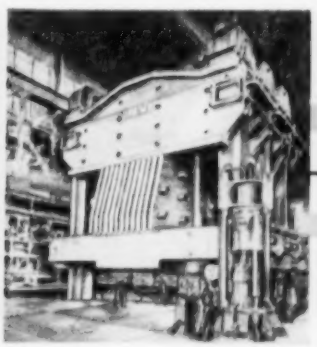

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